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MICROACTIVITY - REFERENCE

USER´S MANUAL - V6 -

MICROACTIVITY – REFERENCE
USER MANUAL

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MICROACTIVITY – REFERENCE
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1. INTRODUCTION

The Microactivity-Reference unit is an automated and computer-controlled laboratory reactor for catalytic microactivity tests. Possible unit configurations:

BASIC UNIT (ATMOSPHERIC PRESSURE)
- Tubular reactor by Autoclave Engineers with 2 μm porous plate. $T_{max.}$= 700 ºC. Thermocouple in catalytic bed, without thermowell. - Reactive system integrated within hot box. $T_{max.}$= 170 ºC. - 6-port VICI valve for reactor bypass. - 3 Hi-Tec Bronkhorst mass flow controllers. - Liquid – gas condenser/separator tank cooled with Peltier cell. - Safety system integrated within microprocessor. - 2 temperature control loops. - 6 control devices for mass flow controllers. - Pressure sensor (0 – 1 bar). - Operating pressure in basic unit: 1 bar. - Equipment design pressure: 100 bar. - Piping, valves and other devices in 316 stainless steel with low dead volume. - Software Process@ for monitoring and data acquisition with distributed control. Remote control via Ethernet. - Heater on gas output line for up to 250 ºC.

PRESSURE CONTROL	LEVEL CONTROL	EXTRAS
- Pressure control system, consisting of a servocontrolled micrometric regulating valve with stepper motor of 1° accuracy . - $P_{max.}$= 100 bar. Accuracy ± 0.2 bar. - Control loop and 100 bar pressure transducer. - Digital communications.	- Liquid/gas separator with level control, consisting of a micrometric regulating valve and capacitive level sensor of low dead volume (0.3 ml). - Control loop and capacitive sensor. - Digital communications.	- HPLC Gilson liquid pump, 400 bar, 0.01 – 5 ml/min. - Space for up to 6 MFC. - Balance at liquid output. - Mass flow meter at gas output. - Special dimensions and materials of reactor. - Extra VICI-VALCO valve for special purposes.

1.1 USING THIS MANUAL

To ensure the correct use and operation of the Microactivity-Reference unit, it is advisable to proceed as follows:

- Read the general description of the equipment in chapter 2.
- Install the instrument as shown in chapter 3.
- Read the description of the equipment's components in chapters 4 and 5.
- Following the operating instructions outlined in chapter 6.

1.2 SAFETY INFORMATION

1.2.1 SAFETY INFORMATION

This unit meets the following EN 61010-1:2001, and it has been designed and tested in accordance with recognized safety standards and designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired.

Whenever the safety protection of the Microactivity-Reference unit has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified servile personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard. Disconnect tha AC power cord before removing covers.

1.2.2 SAFETY SYMBOLS

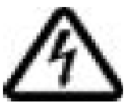
Warnings in the manual or on the instrument must be observed during all phases of operation, service and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Process Integral Development Eng & Tech assumes no liability for the customer's failure to comply with these requirements.

1.2.3 WARNING AND CAUTION CALLS

WARNING: A warning calls attention to a condition or possible situation that could cause injury to tha user.

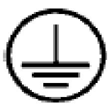
CAUTION: A caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work.

See accompanying instructions for more information



Indicates hazardous voltages

Indicates a hot surface



Indicates earth (ground) terminal

1.2.4 ELECTROMAGNETIC COMPATIBILITY

This device complies with the electromagnetic compatibility requirements subject to the EN 61326:1997 regulation. Operation is subject to the following two conditions:

1. This device may not cause harmful interference.

2. This device must accept any interference received, including interference that may cause undesired operation. If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

   a. Relocate the radio or antenna.

   b. Move the device away from the radio or television.

   c. Plug the device into a different electrical outlet, so that the device and the radio or television are on separate.

   d. Make sure that all peripheral devices are also certified.

   e. Make sure that appropriate cables are used to connect the device to peripheral equipment.

   f. Consult your equipment dealer, Process Integral Development Eng & Tech, or an experienced technician for assistance.

   g. Changes or modifications not expressly approved by Process Integral Development Eng & Tech could void the user's authority to operate the equipment.

2. DESCRIPTION OF THE EQUIPMENT

2.1 GENERAL DESCRIPTION

As may be observed in the P&I diagram shown in Figure 2-1, the system consists of a fixed-bed tubular reactor, with the catalyst bed placed inside upon a porous plate. The flow inside the reactor is up-down, whereby the reactant mixture is fed through the upper part of the reactor and the reaction products are obtained through the lower part.

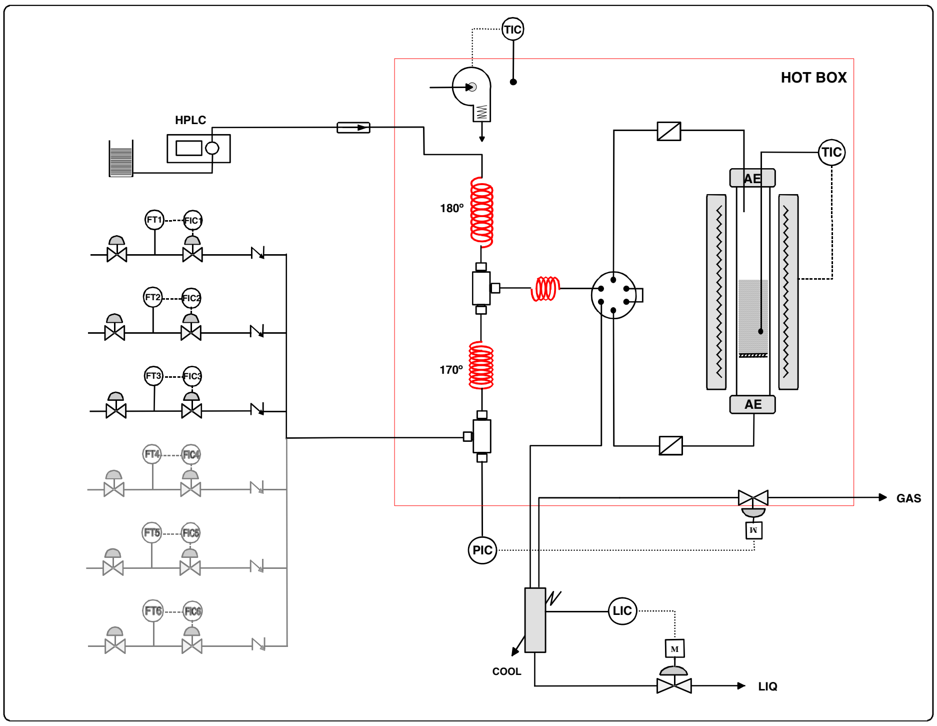


Figure 2-1

After passing through a line shut-off valve, the reactant gas streams are fed into the reactor by means of a system of mass flow controllers that provide a known and controlled flow of gases. In order to stop the products flowing back through the lines, the controllers are protected with check-valves fitted with Kalretz elastomer seals (elastomeric Teflon).

When operation involves liquids, these are dosed by means of a HPLC alternative positive displacement pump, made by GILSON, in streams ranging between 0.01 and 5 ml/min and pressures of up to 600 bar. The liquids are introduced into the system through a low dead volume check-valve.

Liquid and gaseous flows are introduced into the hot box system that includes an electric forced convection heater that allows the process route to be kept at temperatures of 160ºC, and even 180 ºC, to avoid possible condensation in the system. The liquids evaporator is under the heater's forced flow, at temperatures of around 15 to 20 ºC above the rest of the hot box, and the pre-heater for the gases is also under the direct flow of the heater, at temperatures of 10 to 15ºC above the rest of the system.

Once the gases have been preheated and liquids evaporated, these streams merge and flow to a 6-port valve. This valve is operated by remote pneumatic control through the computer or by means of the touch screen and allows for selecting from two possible alternatives for the flow path: either towards the reactor or rerouting it towards the system's gas outlet (by-passing the reactor).

When the flow of reactants is directed towards the reactor, it passes through 10 μm sintered filters made of 316 stainless steel, at both the inlet and outlet of the reactor, thereby protecting the arrangement of valves from possible finely-separated catalyst particles.

At the reactor outlet, and after passing through the 6-port valve, the reaction products pass out of the hot box to the liquid-gas separator, that may be fitted with a high-resolution capacitive level sensor. This system allows the condensation of liquids at low temperature. In the standard series unit, the liquids accumulate inside the condenser and need to be removed manually by the user. If the option has been chosen that includes the level control system in the separator, this removal is performed automatically, providing samples of reaction liquids within extremely short periods of time without accumulation or dilution over time.

The upper part of the separator features the outlet for gases, which are reintroduced into the hot box and are directed to the pressure control system, consisting of a servo positioned micrometric regulating valve that registers the same temperature as the hot box and which provides a continuous and constant flow of gases at the outlet. In those systems that are not fitted with the optional pressure control, this flow goes straight to the outlet.

Once pressure control has been performed, the flow of reaction gases is directed out of the hot box for subsequent measurement and/or analysis by means of a system of, for example, chromatography in gaseous phase.

The Microactivity-Reference unit is fitted with a system of local control and remote control based on communications via Ethernet by means of the Process@ control application. The equipment's safety system is integrated within a microprocessor that is separate from the computer. Accordingly, the alarm signals from the various control loops are centralised in the microprocessor, which operates as programmed to do so with respect to the system's different alarm situations. These actions are triggered on a self-contained and immediate basis, independently of the communications with the computer, thereby upholding the system's safety, as it not only continues operating in the event of failure in the computer system but, in addition, its safety system remains operative. The operation and configuration of the safety system are described later on in this manual.

2.2 SPECIFICATIONS

Equipment	Microactivity - Reference	
Voltage	230 VAC (±5%)	
Frequency	50 Hz (±1%)	
Maximum power consumption	2000 W	
Protection	10 A circuit breaker	
Maximum power consumption of Furnace	800 W (basic unit)	
Maximum power consumption of Hot Box heaters	4 heaters of 165 W	
Remote control interface	Ethernet	
EMC	B class	EN 61326:1997 EN 61010-1:2001
Ambient temperature range for operating	5 – 40ºC	
Ambient temperature range for storing	-20 – 70ºC	
Recommended humidity range	5 – 80%	
Refrigeration	Forced ventilation or convective ventilation	
Dimensions, cm (Height × Width × Depth)	70 × 60 × 55 (Basic Unit)	

2.3 OPERATING CONDITIONS

The optimum operating conditions for the Microactivity-Reference Unit are as follows:

- Pressure: Atmospheric - 100 bar (if the high pressure option is included)
- Temperature: Ambient - 700 ºC.
- Feed: Liquids and gases.
- Flow of reactants: 0.01 – 100 VPH (volume of load per unit of catalyst and per hour) for liquids and 10 – 100,000 for gases.
- Solid catalyst (spheres, pellets, extruded items, etc.)

2.4 EQUIPMENT APPEARANCE

The Microactivity-Reference consists of (see Figure 2-2):

- An integrated unit, whose interior houses the hot box and the reaction system, as well as all the system's control elements and valves.
- A Gilson HPLC pump (optional) for feeding liquids into the reactor.
- A PC with a remote control system involving communications via Ethernet.

At the outlet for reaction gases, the user may incorporate an in-line gas analysis system, which will permit accurate monitoring of the reaction.

2.4.1 GENERAL VIEW

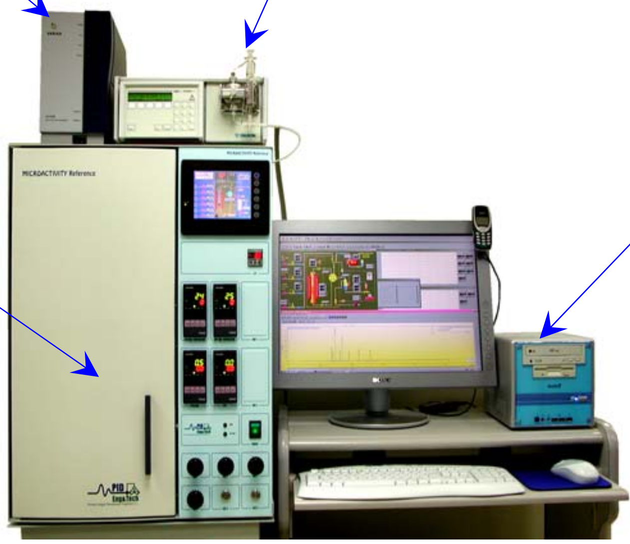


Figure 2-2

2.4.2 FRONT VIEW

Figure 2-3

2.4.3 INSIDE VIEW

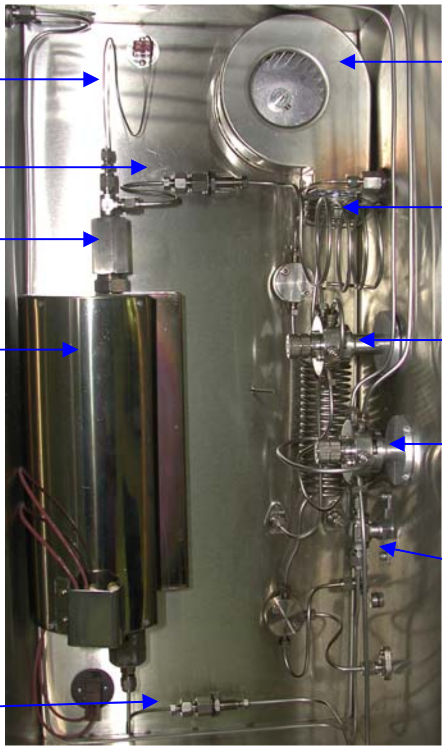


Figure 2-4

2.4.4 REAR VIEW

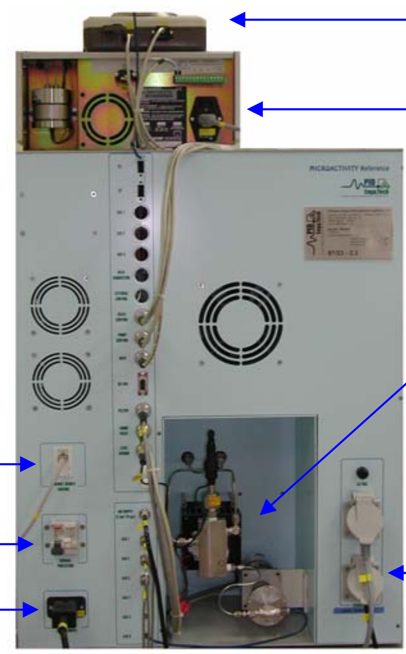


Figure 2-5

2.4.5 INSIDE VIEW - ELECTRONICS

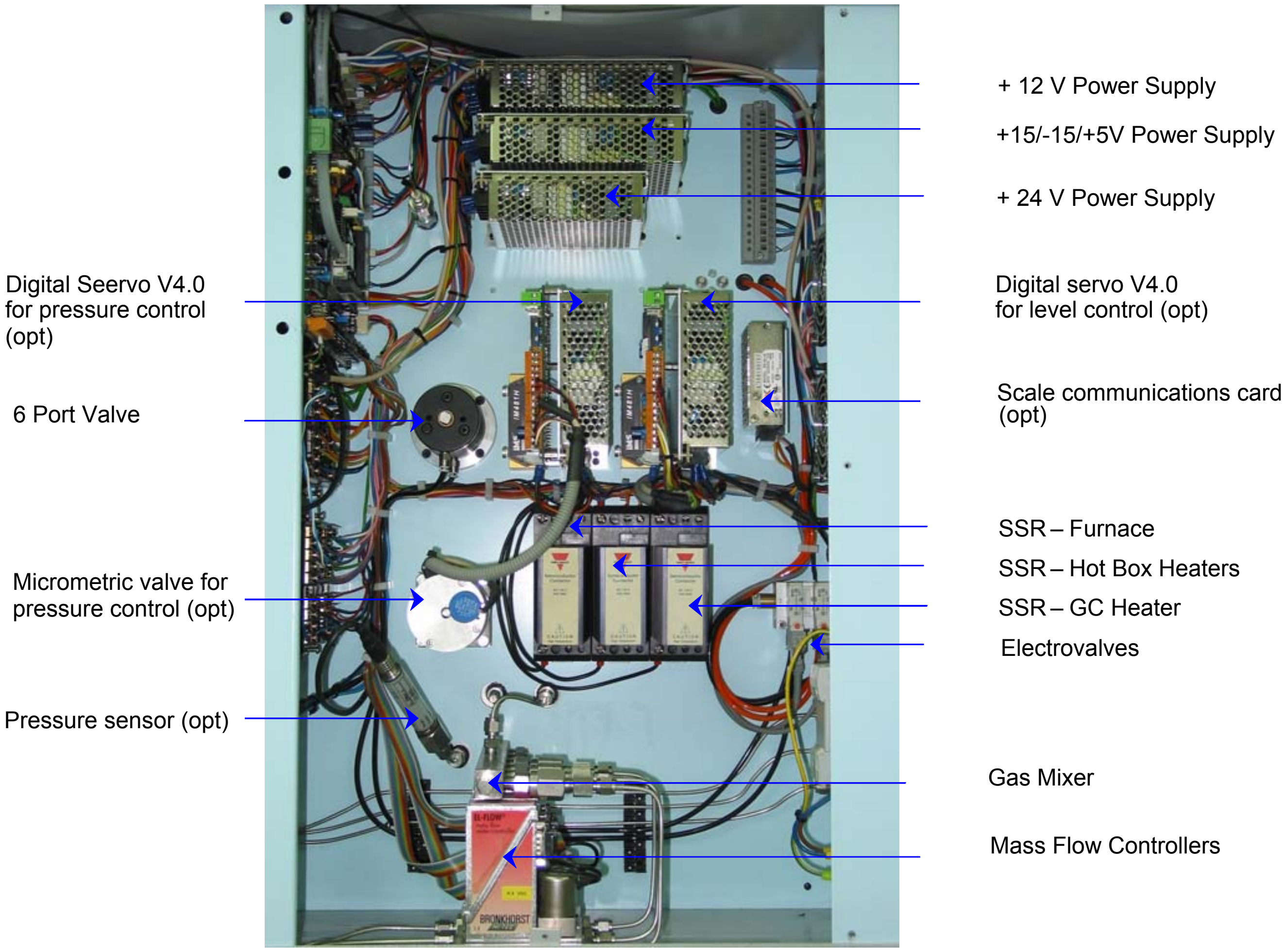


Figure 2-6

3. INITIAL INSTALLATION

3.1 BEFORE STARTING

Before the equipment arrives, make sure your laboratory meets the following environmental, weight, power, and gas requirements. You can find more site preparation information in this chapter.

Site Preparation Checklist:

❒ The site is well ventilated and free of corrosive materials and overhanging obstacles.

❒ Site temperature is within the recommended range.

❒ Site humidity is within the recommended range.

❒ Bench space is adequate for the equipment.

❒ Bench can support the weight of the equipment.

❒ Power receptacle is earth grounded.

❒ Electrical supply meets all equipment's power requirements.

❒ Voltage supply is adequate for oven type.

❒ Gas supplies meet the requirements of the equipment.

❒ Gases and air supply meet the pressure requirements and have two-stage pressure regulators installed.

3.1.1 TEMPERATURE AND HUMIDITY RANGES

Operating the unit within the recommended ranges insures optimum instrument performance and lifetime.

Recommended temperature range	18 – 25 ºC
Temperature range	5 – 40 ºC
Recommended humidity range	50 – 60 %
Humidity range	5 – 80 %
Recommended altitude range	Up to 2000 m

After exposing the unit to extremes of temperature or humidity, allow 15 minutes for it to return to the recommended ranges.

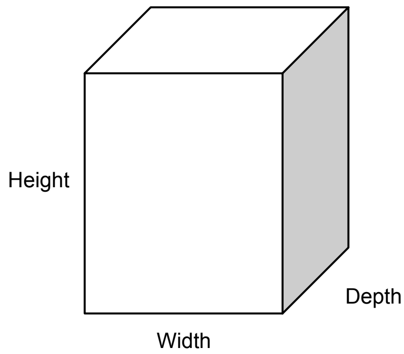
3.1.2 VENTILATION REQUIREMENTS

Do not obstruct air flow around the instrument.

3.1.3 BENCHTOP SPACE REQUIREMENTS

The equipment dimensions are the following:

Height:	70 cm
Width:	60 cm
Depth:	55 cm



3.1.4 ELECTRICAL REQUIREMENTS

3.1.4.1 Grounding

CAUTION: A proper earth ground is required for MA-Ref operations.

To protect users, the metal instrument panels and cabinet are grounded through the three-conductor power line cord in accordance with International Electrotechnical Commission (IEC) requirements.

The three-conductor power line cord, when plugged into a properly grounded receptacle, grounds the instrument and minimizes shock hazard. A properly grounded receptacle is one that is connected to a suitable earth ground.

Proper receptacle grounding should be verified. Make sure the unit is connected to a dedicated receptacle. Use of a dedicator receptacle reduces interference.

CAUTION: Any interruption of the grounding conductor or disconnection of the power cord cause a shock that could result in personal injury.

3.1.4.2 Line Voltage

The unit is designed to work with a specific voltage; make sure your lab has the appropriated voltage option for the unit. The voltage requirements for the equipment are printed near the power cord attachment:

- Voltage:	220 VAC (± 5%)
- Frequency:	50 Hz (± 1%)
- Max. power consumption:	2000 W

3.1.5 GAS REQUIREMENTS

Make sure your lab has the appropriated gas installation for working with the unit:

- Air supply: 5 bar

- Gases: Depending on the unit configuration. The pressure of each reactant gas in the installation must be higher to the working pressure on the unit, and appropriated to the mass flow controllers that the unit incorporates.

3.2 EQUIPMENT INSTALLATION

Before starting, be sure to have available all the tools and pieces necessary for the installation.

3.2.1 UNPACKING THE EQUIPMENT

Unpack the unit carefully and inspect the shipping containers for damage. If a container is damaged or shows signs of stress, notify both the carrier and Process Integral Development Eng & Tech.

Keep all shipping materials for inspection by the carrier. Check the items received against the packing lists. If there are discrepancies, notify Process Integral Development Eng & Tech immediately. Keep the shipping containers until you have checked their contents for completeness and verified instrument performance.

3.2.2 PLACING THE EQUIPMENT ON THE BENCHTOP

The unit requires a benchtop that can support its weight plus that of other equipment you will use with it. The area must be free of overhanging obstructions that might interfere with cooling and limit access to the top of the instrument.

⚠ **WARNING:** Be careful when lifting the unit. Because it is heavy, two people should lift it. When moving the equipment, be aware that the back is heavier than the front.

3.2.3 ELECTRICAL INSTALLATION

The unit's electrical installation is performed as described forthwith:

⚠ *For reasons of safety, do not connect to the mains until the full installation of the equipment has been completed.*

The installation of the external devices that are described will only be possible if they have been chosen as a configuration option of the Microactivity-Reference reactor.

1. Installation of the liquid – gas separator (Peltier): Connect the end of the cable (power supply) to the "Peltier" round connector that is to be found on the rear of the reactors' hot box, as shown in Figure 3-1:

Figure 3-1

2. Installation of the liquids pump: Before installing the pump, make sure the on/off switch to be found on the rear of the pump is in the off position (o), Figure 3-2:

Figure 3-2

The following connections are to be made (see Figure 3-3):

a. Connect the power cable to one of the two 220 VAC power sockets on the rear of the unit. These power sockets will cease to supply power when the equipment is switched off by means of the switch on the front, so it is not advisable to connect analysis equipment or other devices that need to operate separately from the Microactivity-Reference unit.
b. Connect the connection terminal (stop for system alarm), as well as the sub-D connector (digital communications), on the rear of the pump.
c. Connect the round connector on the end of this same cable to the "**Pump Control**" on the rear of the Microactivity-Reference.

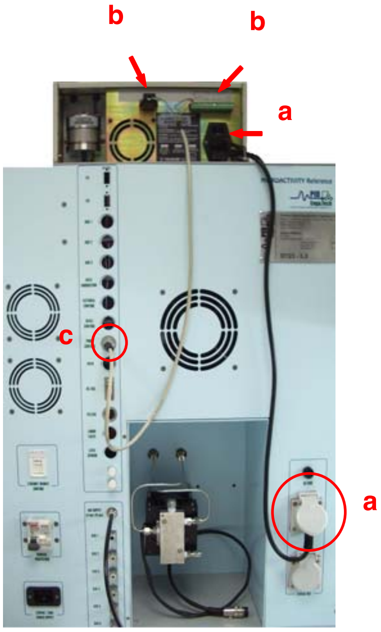


Figure 3-3

3. Installation of the liquid scale (see Figure 3-4):

   a. Connect the sub-D type connector (digital communications) to the rear of the scale.
   b. To the left of the sub-D connector, connect the JACK type plug (power supply).
   c. These two connectors are joined in a single round connector that has to be plugged into the corresponding socket on the rear of the hot box ("**Scale Control**").

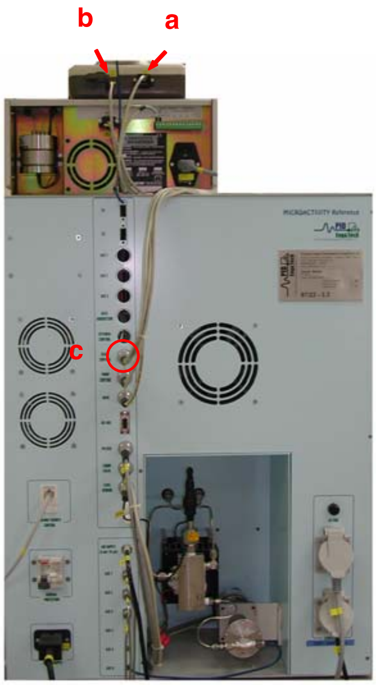


Figure 3-4

4. Other components: On the rear of the Microactivity-Reference's hot box there is a circular type connector for each one of the devices that may be connected to the unit, such as:
    - Mass flow meter ("**MFM**").
    - Valve for regulating the level of liquids in the condenser ("**Liquid Valve**").
    - Sensor for the level of liquids in the condenser ("**Level Sensor**").

5. Gas outlet on the system: Leading to the analysis system. A heater is included for this line (see Figure 3-5):

Figure 3-5

6. Connection to Ethernet: The direct connection between the Microactivity-Reference and the control PC is performed by means of the crossed cable supplied with the equipment, connecting it on the rear of the reactor box ("Ethernet Remote Control", Figure 3-6). When the connection is made via Ethernet, it is made with a category 5 UTP connection cable for Ethernet networks with a RJ45 connector. The Ethernet connection between the PC and the Microactivity-Reference is reduced to 10 Mbts base-T.

Figure 3-6

7. The reactor's power socket is on the lower rear of the reactor (Figure 3-7).

WARNING: Before connecting the equipment's power supply, make sure that the main circuit breaker is in the "OFF" position (Figure 3-8).

Figure 3-7

Figure 3-8

3.2.4 GAS INSTALLATION

Once the electrical installation has been performed, the next step is to install the gases that the Microactivity-Reference unit is going to work with. To do so, all that is required is to connect a 1/8´´ line, preferably of 316 stainless steel, between the pressure reducers on the gas cylinders and the system's gas inlet, which is to be found on the upper part of the equipment (Figure 3-9).

The connection is to be made as follows:

- **Synthetic air**:
  - Inlet pipe: Polyethylene 6×4 mm pipe, supplied with the equipment (3 m).
  - Connection: Quick-fit connection, pressing the pipe against the adapter.
  - Inlet pressure: 5 bar
  - It operates on the pneumatic systems for door and oven opening, 6-port valves, etc.

- **Gases 1, 2,.. etc.**: Reaction gases.
  - Inlet pipe: 1/8´´ pipe in 316 stainless steel.
  - Connection: Gyrolok 1/8´´ adapter.
  - Inlet pressure: 5/10 bar above the operating pressure. The inlet pressure for each one of the gases depends on the mass flow controller installed, whereby its specifications should be consulted before making the gas connection.
  - The gas inlet position (1, 2, etc) depends on the arrangement of the mass flow controllers on the equipment

Air supply

Reaction gases

Figure 3-9

Once the installation has been made, the following steps are to be performed in order to introduce gases into the system:

1. Open the pressurised gas cylinders.
2. Adjust the inlet pressure for each one of the gases by means of the pressure reducers.
3. Open the on/off valves for each gas on the lower part of the front panel.

At this point, the system is ready to operate. Inversely, upon concluding operations with the Microactivity-Reference, proceed as follows:

1. Reduce the inlet flows to zero for each one of the reactant gases.
2. Preferably flush the installation with inert gas for 5 min.
3. Close the on/off valves on the front panel.
4. Whenever possible, close the reactant gas cylinders and reduce the pressure of the pressure reducers to zero (if the system is leak-free, the inlet lines to the system will register the inlet pressure). In the case of compressed air, the installation is to remain permanently pressurised in order to permit the pneumatic operation of the valves and the door on the hot box.

3.2.5 TURNING THE POWER ON

Verify that the power switch is in the OFF position (o). Plug the power cord into the power receptacle. Put the circuit breaker in ON position and turn the power on with the frontal switch (position I).

4. COMPONENTS OF THE MICROACTIVITY-REFERENCE UNIT

Figure 4-1 shows a diagram of the items that constitute the Microactivity-Reference reactor's arrangement of lines and instrumentation, including 3 mass flow controllers for the gas inlets, as well as the pressure control options in the reactor and liquid level control in the liquid – gas separator.

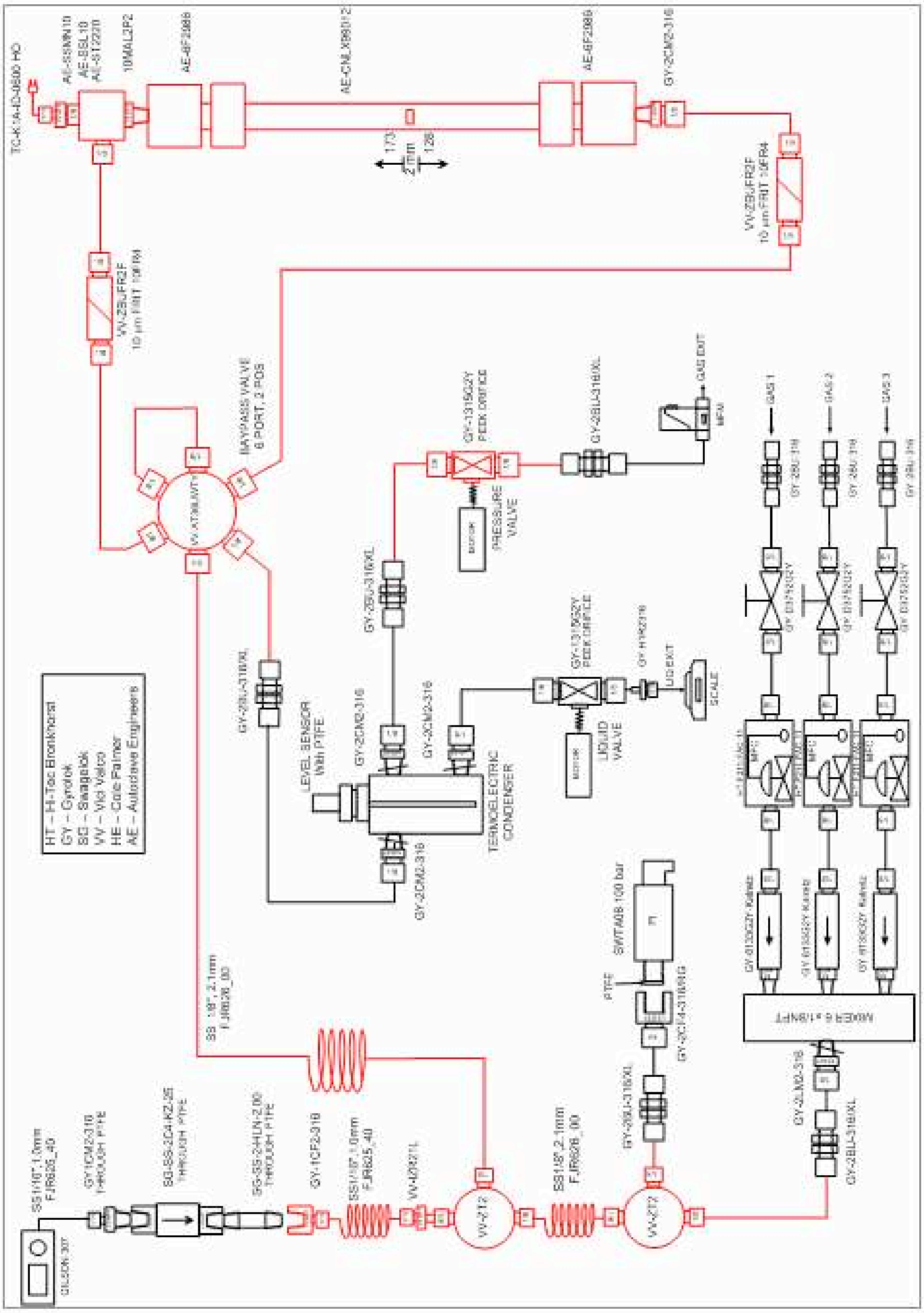


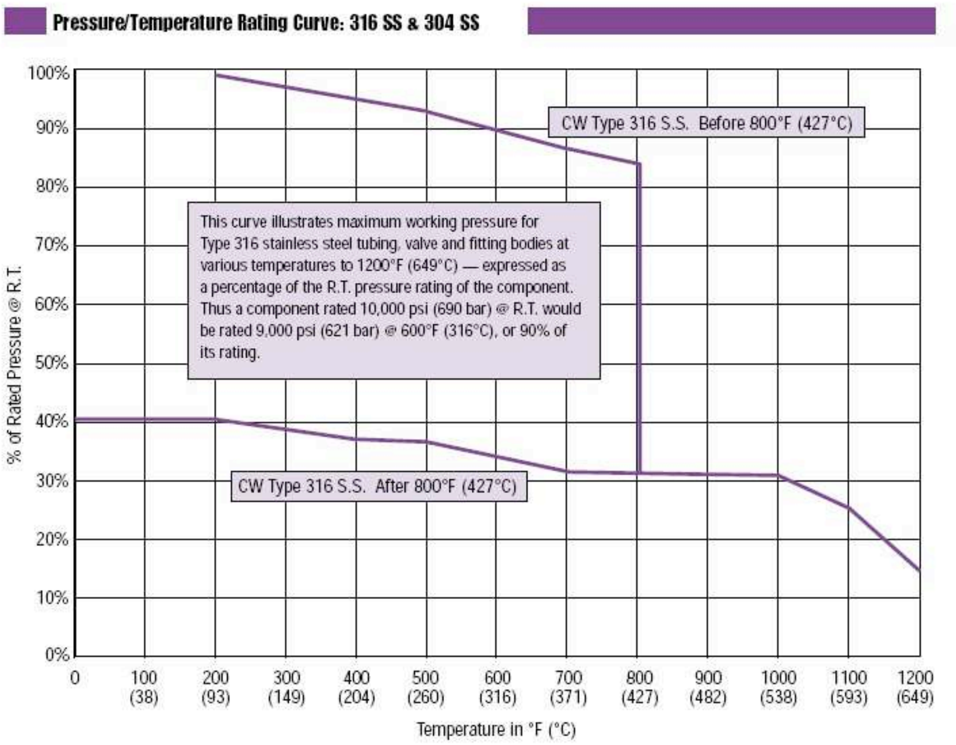
Figure 4-1

4.1. THE REACTOR AND THE HOT BOX

The tubular reactor consists of a nipple made by Autoclave Engineers, model CNLX99012, whose standard model has the following specifications:

- Length: 305 mm
- External diameter: 14.5 mm
- Internal diameter: 9 mm
- Material: 316-L Stainless steel
- Internal volume: 20 ml
- Connections: SF562CX
- Seals: AE-6F2986
- $T_{max.}$ recommended: 700ºC
- $P_{max.}$ recommended: 1350 bar at 25ºC; 400 bar at 482ºC (P$_{max}$ reactor: 100 bar)

The following graphic illustrates the maximum working pressure for type 316 stainless steel nipple of Autoclave Engineers depending on the temperature.



Depending on the user's requirements, any other reactor size (diameter or length) or any other construction material (Monel 400, Inconel 600, Titanium Grade 2, Nickel 200, Hastelloy C276, Titanium 6AL4V, etc) can be used.

As may be seen in Figure 4-2, the inside of the reactor has been fitted with a 10FR4HC porous plate made by the firm VALCO, in Hastelloy C-276 with a pore size of 2 μm. This plate rests on a 316 stainless steel pipe inserted through the lower end of the reactor, thereby allowing for:

- Reducing to a minimum the dead volume at the outlet
- Replacing the plate whenever so required, without the need to replace the reactor nipple.

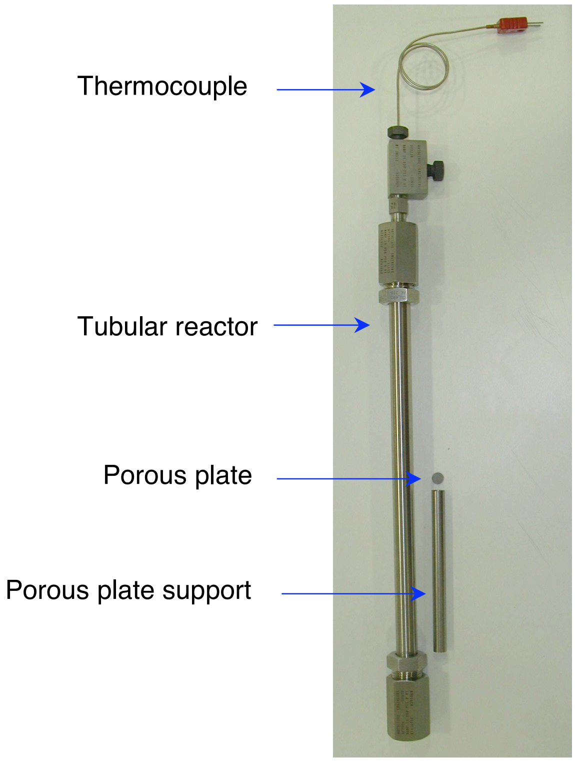


Figure 4-2

The thermocouple, type K (encased in a 1.5 mm diameter Inconel sheath), in inserted through the upper end and is in contact with the catalyst bed without thermowell. This allows for reading reaction temperatures with response times in milliseconds.

The reactor is housed in an oven built without insulation, consisting of a 304 stainless steel chassis, with the inside housing the resistance together with a refractory material:

- 800 W / 220 VAC
- Maximum operating temperature: 800ºC.
- Low thermal inertia.
- Automatic opening system with temperature warning system.

The entire system is contained within a hot box made of 304 stainless steel, whose interior holds an electric convection heater. Its maximum recommended operating temperature is 190ºC.

4.2. THE CONTROLLERS

4.2.1. THE REGULATION PARAMETERS

The Microactivity-Reference unit uses P-I-D controllers for the following control loops:

- *Control of reaction temperature:* The signal from the thermocouple located in the catalyst bed is assessed by the controller, whose output signal is relayed to a solid-state zero-switching relay that regulates the power supplied to the oven proportionally to the control signal. The power the oven receives corresponds to a signal between 0 and 200 VAC, typically between 0 and 140 VAC, proportional to the control signal.

- *Control of hot box temperature*: The signal from the thermocouple located inside the hot box is assessed by the controller, whose output signal is sent to a relay that regulates the power supplied to the box's heater proportionally.

- *Pressure control:* The signal from the pressure transmitter installed upstream of the reactor is assessed by the controller, whereby its output signal determines the position of the pressure control valve.

- *Level control:* The signal from the capacitive level sensor installed in the liquid – gas separator is assessed by the controller, whereby its output signal determines the position of the level control valve located in the base of the separator.

The controllers used are made by the firm TOHO, model TTM-005, catering for RS-485 digital communications, and are shown in Figure 4-3:

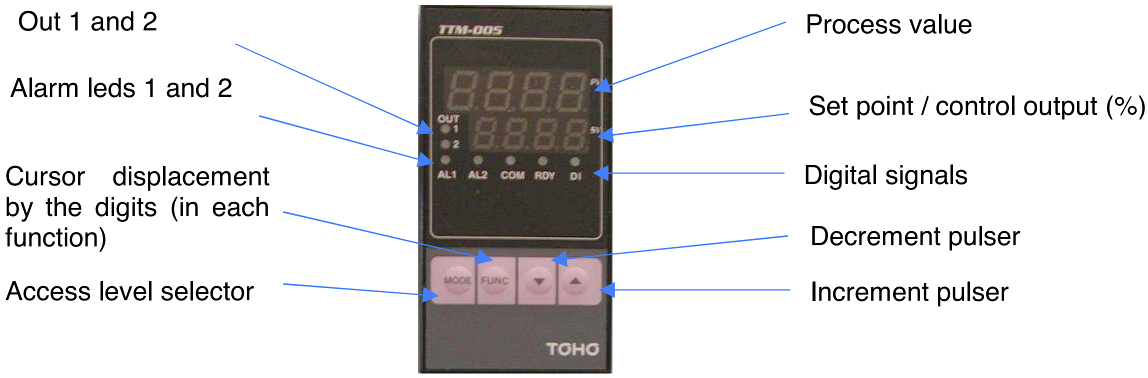


Figure 4-3

The process value of the controlled variable is displayed in the upper window on the controller (green), whereas the set-point or the % control output (depending on whether operation is in automatic or manual mode) is displayed in the lower window (red). This set-point may be changed by pressing the "**Func**" key, which allows for selecting each one of the different digits and, subsequently, changing the value of each digit using the ▲and▼ keys. The "**Mode**" button is used to access the different control parameters configured in the controller:

- **-** : Process value.
- _ **P**: Proportional band.
- _ **I**: Integral action (s).
- _ **d**: Derivative action (s).

- _ **nd**: Control mode. Select with the ▲and▼ keys:
    - *Run*: Automatic mode. The user is to set the desired value or set-point of the variable controlled on the lower screen of the controller, which will automatically act on the variable's control output.
    - *Rdy*: Control start at a given moment. When this mode is activated, the red LED "RDY" on the front of the controller is lit up.
    - *Man*: Manual mode. The user is to set the variable's control output (e.g. heating power: 0 – 100%) on the lower screen of the controller.

  As a general rule, the user has to operate under *RUN* mode (when the variable's set-point is set) or *MAN* (when the control output is set), but never under *RDY* mode (the LED "RDY" on the front is to remain off).
- _ **nu1**: The variable's control output (%). This parameter will be modified by the user whenever operating under manual mode (never under automatic).
    - Regarding the temperature control, this parameter indicates the % of heating of the heater.
    - Regarding the pressure and level controls, this parameter indicates the % of opening of the control valve (fully closed at 0% and fully open at 100%).

    The parameter "_ **AH1**" on the **SET 2** set-up menu represents the maximum control output for the controller (access this menu by depressing the "**Mode**" key for 2 sec., press the "**Func**" key and enter a 2 in the cursor using the ▲ key. Move through the different menu parameters by pressing "**Mode**" until the desired parameter is reached).
- _ **E1H**: Upper alarm limit. Value of the variable above which the system's alarm is to be triggered.

Although these parameters have already been set with their optimum control values for operation of the Microactivity-Reference unit, they may be modified whenever necessary by using the "**Func**" button to go to each one of the digits and then use the ▲ and ▼ keys to increase or decrease the value.

The standard set-up for the Microactivity-Reference reactor is detailed in Table 4-1:

Control parameter	Reactor temperature	Hot box temperature	Pressure	Level
_ P	24.0	9.0	25.0	110.0
_ I	180	120	90	50
_ d	20	20	0	0
_ nd	run	run	run	run
_ nu1	-	-	-	-
_ EH1	700	40 (do not modify)		

Table 4-1

Other parameters of major importance that configure this type of controller are those that are shown below in Table 4-2, with their standard values for operation with the Microactivity-Reference unit (these parameters may vary from one unit to another. For verification of a reactor's specific set-up, consult the technical documentation that is supplied with the equipment):

		Reactor Temperature	Hot Box Temperature	Pressure	Level	Aux 1	Aux 2
	LnP	0	0	22	22		
	PuG	1.00	1.00	1.00	1.00		
	PuS	0	0	0,9	0.00		
SET1	PdF	1	1	1	1		
	DP	0	0	0.0	0.00		
	Fu	1	1	1	1		
	LoC	0	0	0	0		
	SLH	800	200	250	10.00		
	SLL	0	0	0.0	0.00		
	Nd	run	run	run	run		
	Cnt	110	110	110	110		
	DIr	0	0	1	1		
	Nul	-	-	-			
	TUn	1	1	1	1		
	AtG	1.0	1.0	1.0	1.0		
SET2	AtC	2	2	2	2.00		
	P1	45	16.0	25.0	110		
	I	550	120	50	50		
	D	20	20	0	0		
	T1						
	ArW	100.0	100.0	100.0	100.0		
	MH1	80	95.0	85	85.0		
	ML1	0.0	0.0	0.0	0.0		
	Pbb	0.0	0.0	0.0	0.0		
	E1F	6	6	6	6		
	E1H	700	40	100.0	2.00		
SET3	E1C	0	0	0.0	0.00		
	E1t	0	0	0	0		
	E1b	0	0	0	0		
	E1P	0	0	0	0		
	Con	b8n1	b8n1	b8n1	b8n1		
	bPS	19.2	19.2	19.2	19.2		
SET6	Adr	1	2	4	5		
	AWt	0	0	0	0		
	nod	ry	ry	ry	ry		
SET7	tno	0	0	0	0		
	rP1	0.0	0.0	0.0	0.0		
	Pr11	P1	P1	P1	P1		
	Pr12	I	I	I	I		
	Pr13	d	d	d	d		
	Pr14	Nd	Nd	Nd	Nd		
SET0	Pr15	Mu1	Mu1	Mu1	Mu1		
	Pr16	E1H	OFF	E1H	E1H		
	Pr17	rP1	OFF	OFF	OFF		
	Pr18	OFF	OFF	OFF	OFF		
	Pr19	OFF	OFF	OFF	OFF		

Table 4-2

P-I-D values depends on the operation conditions. As a example, in Table 4-3 are shown the PID parameters for the reactor oven, depending on the working temperature:

	Temp Reactor	
	200 - 400 ºC	500 - 600 ºC
P1	42	30
I	465	350
D	15	15
MH1	80	50

Table 4-3

There now follows a brief introduction to the different methods that are used for tuning proportional, integral and derivative (PID) controllers and the criteria that are followed for considering that optimum control of the process has been achieved.

4.2.2. CONTROL STABILITY CRITERIA

Stability is the control system feature that makes the variable return to the set-point following a disturbance. The most commonly used criteria for determining control stability are the following:

A) Criterion of minimum area or of damping ratio

This is the criterion of widest application, especially regarding processes in which the duration of the deviation is as important as the value of the same. According to this criterion, the control is to ensure that the area of each oscillation in the control output signal following a disturbance is minimum (experience shows that stability criteria should be applied onto this signal and not on the process variable). In other words, to achieve a minimum error in the shortest time possible.

Experience in industrial control processes indicates that this area will be minimal when the proportion between the peak-to-peak amplitudes of the first two consecutive cycles, immediately following the disturbance, is 1/4. In other words, the damping ratio between these consecutive peaks must be 25%. It is a compromise criterion between stability in the controller's response and the speed or rapidity with which the manipulated variable returns to a stable value:

- Proportions higher than 25% give greater stability, but they lengthen the time required for attaining stationary state.
- Proportions lower than 25% may reduce the time it takes to reach stationary state, but they cause instability in the system.

B) Criterion of minimum amplitude

The control system is to keep the amplitude of the deviation to a minimum. It is used in processes in which the equipment may be damaged by sudden large-scale deviations. For example, in reactions with thermal self-ignition, equipment featuring rupture discs, etc.

C) Criterion of minimum disturbance

The control system is to provide a non-cyclic recovery curve, precisely to ensure that the cyclic variants do not disturb or influence other system processes. This situation is forthcoming in concatenated processes, in which the oscillations in one subsystem are the result of oscillations in others. In a case like this, the decision must be taken to overdamp the control systems or perform the start-ups as per manual procedures.

4.2.3. CONTROLLERS TUNING

The values of the proportional band (inverse to the gain), integral action time (seconds / repetition) and derivative action time (seconds of advance) need to be conveniently dovetailed with all the other elements in the control loop so that, in the event of a disturbance in the system, the latter's response fulfils the control stability criterion.

The adjustment systems are classified into two categories:

- Experimental methods: Applied when the process model is unknown. They determine the process's static and dynamic characteristics on the basis of one of several measurements obtained from the real process. The two most frequently used for closed

control loops are: the trial and error method (test-error-test) and the ultimate gain method, developed by Ziegler & Nichols.

- Analytical methods: Applied when the process model or the equation relative to the system's dynamics are known. They are difficult to apply in pilot plant control systems, given the absence of reliable data on the processes, and they are only applied when sufficient information is available for the perfect identification of the process model (transfer function), usually in industrial environments.

It is worth noting here that the procedures referred to as auto-tuning are based on empiric experiences and results obtained in industrial environments, which have nothing to do with the processes taking place in a laboratory pilot plant.

4.2.3.1. Ziegler & Nichols method

It is the most widespread experimental method for tuning the regulation parameters of a PID controller, although it is not recommended when the tuning is carried out mainly with a view to stable transition between different process states (variations in reaction temperature for the screening of catalyst activity at different temperatures) instead of seeking the long-term stability of the same.

It allows for calculating the three values of the PID actions on the basis of the data obtained in a quick test of the characteristics of the closed control loop.

In short, it consists in gradually narrowing the proportional band from an initial value (e.g. 15 %) with I=0 and D=0 whilst small disturbances are created, until the process begins to oscillate continuously. This value of P receives the name of "ultimate proportional band" or "critical proportional band", Pc. Measurement is now made of the period of these oscillations (Tc, in seconds), that is, the time that elapses between two consecutive oscillations when the system is at its critical proportional band.

The controller parameters that will produce a response with the 25% damping ratio are calculated as per:

Proportional band (%)= 1.5 · Pc
Integral action (s)=0.5 ·Tc
Derivative action (s)= 0.1 · Tc

The optimum selection of the controller parameters is always a compromise solution and one that depends on the skill of the operator. Thus, for a process in which there is a considerable transport delay, it will be advisable to use high values of the proportional band. On the other hand, high values of P imply considerable sluggishness in the system's response to external disturbances or those of the system itself.

Typical values in semi-industrial processes for the P, I and D control parameters are:

Fast systems (pressure, flow):	P = 0 – 25% I = 1 – 120 s D = 0 – 10 s
Slow systems (temperature):	P = 0 – 50% I = 60 – 600 s D = 2 – 60 s

4.2.3.2. Method of trial and error

This is carried out with the controller and the process operating in standard mode. The general procedure basically involves starting up the process and performing repetitive tests on each control action (beginning with the proportional band), introducing disturbances by changing the set-point and returning to its initial value. The adjustment is gradually fine-tuned by observing the control output signal and its response to the disturbance created (not too severe, so as to avoid damage in the process).

Begin by observing the system's behaviour in on-off status. A proportional action is then generated whereby the oscillations are suitably attenuated. Begin with a wide proportional band (small gain), which is then gradually narrowed, in line with the system's evolution, until the required stability is attained (damping ratio of 25% between two successive waves).

Once a suitable value of parameter P has been achieved (compromise between stability and error offset), the elimination of the offset will be achieved by means of the addition of an integral control action, also by trial and error.

As integral action compromises control, the proportional band is to be raised slightly (lower gain) and, beginning with a high value for the integral action (in seconds), slowly decrease it, whilst at the same time creating disturbances in the process by means of changes to the set-point.

Once the proportional and integral action parameters have been tuned, the derivative action is increased in small jumps, from D=0, whilst at the same time creating disturbances in the process by means of changes to the set-point, until the process obtains its characteristic cyclic behaviour. A suitable value for the derivative action should lead to the stabilisation of the controlled variable a few cycles after a disturbance.

4.2.3.3. Method proposed by PID Eng & Tech

Based on the accumulated experience of PID Eng & Tech in the tuning of pilot plant or laboratory processes, where the system gains are high (transport delays are lower than normal in industrial environments) and where the system's readiness to respond to changes in the set-points is especially prevalent, a new method has been designed for tuning said parameters, based on the experience acquired in the control of processes.

It is relatively easy to predict the value of the proportional band that is suitable for a process if one bears in mind the physical interpretation of this concept. If the proportional band located around the set-point is understood to be the area within which the controller goes from providing a control output of 0 to 100%, and if manual manipulation has been made beforehand of the final control element in the process conditions, the operator may know, for example, that in a pressure control system, the valve must remain closed until a pressure of 86 bar is achieved when the aim is to reach 90 bar, and as of that moment, the control action may be performed to regulate the set-point. This indicates that the proportional band should have a value of 8 bar (4 above and 4 below the set-point). If it is taken into account that the operating interval is 100 bar, this 8 bar proportional band corresponds to a value of P = 8% (% F.S.).

If this is the first time this process is initiated, precautions may be taken such as increasing this value with a view to overdamping the system and, in addition, carrying out the system's first start-up below a hazardous position, remembering that the offset in this system is unknown, and may equally be positive or negative.

The application of this procedure to any kind of system may allow for foreseeing the suitable value of P by simply sensing when the controller should begin to change its control outlet so as not to overrun the order.

An interesting possibility for advanced operators is to perform this initial trial and error on the value of P with a high value of integral action, which will avoid the offset phenomenon without affecting the stability of the solely proportional action. This high value of integral action should correspond to, for example, 2 or 3 values of the oscillation period, which for rapid systems (pressure, flow, level, etc.) will correspond to 20 - 60 s, and for slow systems (temperature, pH in buffered solution, etc.) to 200 - 600 s. It tends to be relatively easy to deduce a system's period of oscillation by bearing in mind the characteristics of the same.

Once the system has been started up with this estimated value of P, in all probability following one or two trial runs around the set-point, the value of Pc is found, whereupon the critical oscillation period will also be known.

As has been noted on several occasions, pilot plant operation involves a scanning of different conditions around the operating variables. Given that this is the case, it is bad practice to select a value of P similar to that of Pc, given that a modification in the set-point, or a modification involving another operating variable, may suddenly change the system's gain and destabilise it (if the value of P remains below the value of Pc).

Accordingly, as with what is recommended in other empirical methods, an appropriate value for P in these systems may be:

$$P = 1.6 \cdot Pc$$

Concerning the appropriate value of I, the procedure in which this control action operates may be understood as follows: the control algorithm assesses the area comprised between the variable's oscillation curve and the straight line delimited by the set-point. Accordingly, if the time in which the totalling of this area coincides with the period of oscillation, its positive part is cancelled out by the negative and the result, for the action that is superimposed onto the proportional action, is zero.

If this operation is performed in a short period of time, the result will not reflect the true situation and the resulting control action will destabilise the control system. On the other hand, if this assessment is performed over the course of two or three cycles, the result will continue to be zero, with the singularity that too long will have been spent waiting to undertake an action that would have corrected the error earlier, a situation that is clearly never desirable. Therefore, low values of I are damaging and high values of I, albeit not damaging, are not convenient.

But if the value of I in these pilot plant systems is adjusted to the period of oscillation, the situation resulting from a significant change in a process variable or set-point in which the system's gain becomes more pronounced and, therefore, a change occurs in the system's period of oscillation, could lead to process instability, as the time spent in calculating the area of this new situation has not been sufficient to allow for the compensation of the positive and negative areas of this oscillation.

Thus, for this type of systems, it is advisable to select a value for parameter I that is higher than the critical oscillation period (Tc):

$$I = 1.2 \cdot Tc$$

Concerning the derivative action, and always bearing in mind how problematic its use is for non-advanced operators, the option should be taken not to use it in rapid systems (the gains on

pilot systems are very high due to the immediate response to a disturbance, as a result of their low damping capacity).

For systems that evolve slowly, and due to their nature of “overtaking” the process’s evolution, relatively narrow values improve systems’ response to overshoot phenomena during start-up procedures. Thus, for systems in which these phenomena are frequently repeated during the operating procedure, desirable values may be:

$$D = 0.07 \cdot Tc$$

Clearly, experience will determine the optimum values for the tuning of a control loop, with these recommendations being nothing more than an approximate departure value. It is important to stress that by making use of these parameters, an advanced operator will be able to “draw” a variable’s approach curve to its situation of stability following a disturbance.

4.3. THE LIQUID – GAS SEPARATOR

The Microactivity – Reference unit includes a liquid – gas separator of low dead volume consisting of a stainless steel tank on whose walls liquids condense at high pressure and low temperature. Once they have passed through the reactor, the reaction gases are drawn outside through the rear of the hot box, where the separator is located (see Figure 4-4) and where liquid condensation takes place. Upon leaving the separator, the gases are again introduced into the hot box, flowing to the pressure control system (provided that the unit has this set-up option).

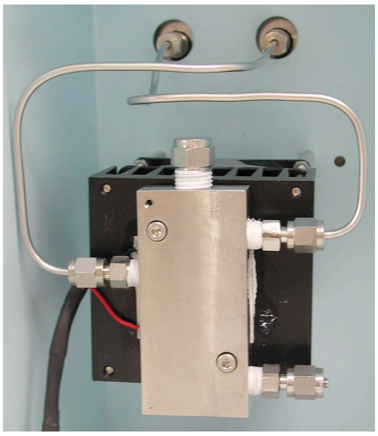


Figure 4-4

The separator consists of a solid piece of 316 stainless steel in which a perforation has been drilled of 65 mm in depth and 8.5 mm in diameter, as well as other machining corresponding to the system's inlet and outlet, and which acts as a condenser by means of a Peltier cell.

A Peltier cell consists of two facing ceramic panels between which there are hundreds of thermoelectric couples. Just as a thermoelectric couple generates a difference of potential when its connections register different temperatures (Siebeck effect), when a difference of potential is applied to the thermoelectric couple, a difference of temperature is generated between the connections (Peltier effect).

The application of a 15 VDC difference of potential and a 3 A current in the Peltier cell generates a temperature difference between the panels of approximately 30ºC. If a forced convection heat sink is used to bring the temperature of the hot panel to 25ºC, then, and to uphold this temperature difference of 30ºC, the temperature of the cold panel must fall below –5ºC, and when placed in contact with a metallic block, temperatures of around 0ºC will be achieved in that block.

The cooling of this tank may be activated in two ways:

- Via the main screen of the touch screen (see section 4.5.1 of this manual).
- Via the process@ control software (see section 5.5.3 of this manual).

Regarding equipment that is not fitted with a level control in the separator, the removal of the condensed liquid is to be performed manually.

4.4. THE PRESSURE SENSOR (FOR EQUIPMENT AT ATMOSPHERIC PRESSURE)

This device is only available in those pieces of equipment that operate at atmospheric pressure, which do not include the pressure control option in the reactor (chapter 4.6.2 in this manual). Its purpose is to register the pressure drop inside the reactor.

APPEARANCE OF THE DEVICE

- 1): Display. It shows the value of the pressure measurement, adjustments, error messages and the keyboard blocking status.
- 2): Indicator that lights up when the operation of comparative action of output 1 has been activated.
- 3): Indicator that lights up when the operation of comparative action of output 2 has been activated.
- 4): Increase button.
- 5): Decrease button.
- 6): Key for selection of operating mode.

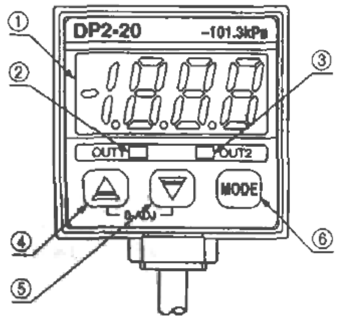


Figure 4-5

SPECIFICATIONS OF MODEL DP2-21

- Range of read-out: 0 – 100 kPa (0 – 1 bar). The application of pressures above the maximum read-out pressure could damage the device.
- Maximum pressure admissible: 490 kPa (4.9 bar).
- Units of measurement: bar (by default in the Microactivity-Reference unit, although these may be modified by the user).
- Fluids applicable: Non-corrosive.
- Response time: ≤ 2 ms.

METHOD OF OPERATION

Once the Microactivity-Reference unit has been switched on, the pressure sensor will at all times display the pressure drop in the system.

SYSTEM ALARMS

By pressing twice the "**Mode**" button, the device's screen displays the upper pressure limit. If this is exceeded, the system's pressure alarm is triggered. The actions to be performed by the system in the event of such an alarm are as defined by the user on the pressure alarm screen on the Microactivity-Reference's touch screen (see section 4.5.2 of this manual).

By pressing once on the "**Mode**" button, the device's screen displays the pressure value below which the system's pressure alarm will be shut off, once the situation that triggered the alarm has been resolved.

ERROR MESSAGES

Message	Cause	Corrective action
E-1	Current surge due to a short circuit.	Reboot the device.
E-3	Pressure is being applied to the device during the adjustment of the zero-setting.	Carry out the adjustment of the zero-setting at atmospheric pressure.
- - -	The pressure applied exceeds the upper limit of the pressure range that can be shown.	The pressure applied must be within the range that can be shown on the screen.
- - - -	The pressure applied exceeds the lower limit of the pressure range that can be shown.	

Table 4-4

For more information on the different operating modes, as well as the different possible configurations for the device, consult the manual for the series DP2 pressure sensor.

4.5. THE TOUCH SCREEN

The Microactivity-Reference unit has a touch screen, which allows for adjusting the various process parameters, displaying the following:

- A main screen, which shows a P&I diagram of the process
- Different set-up screens, which are accessed from the main menu.

4.5.1 MAIN-SCREEN FUNCTIONS

The main screen presents the process flow, and has the appearance shown in Figure 4-6. Pressing on the different icons and buttons grants access to the different functions:

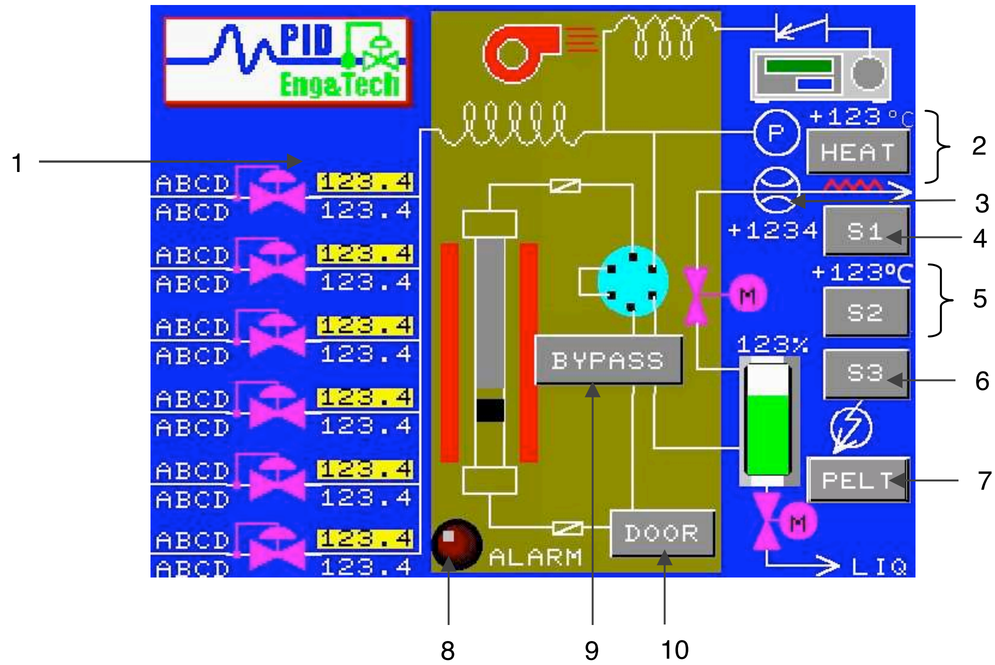


Figure 4-6

Buttons could be in two different states:

- Green background: Activated
- Grey background: Deactivated

1. Control of gas inlet streams: The screen depicts each one of the mass flow controllers integrated within the system, showing:
   - Set-point (yellow background): set by the user. Pressing on the value calls up a numerical keyboard for entering the desired set-point, which is stored in the system by pressing the "**Enter**" key.
   - Current process value (on the lower part): It cannot be modified by the user.

2. **HEAT**: Heating of the system's gas outlet line.
   It is possible to incorporate a thermocouple in this line (opt.), checking the line temperature on the touch screen, over the button. If the unit does not incorporate this option, this temperature reading appears as a line of points. The configuration of this control loop can be set in the screen "**MISC SETUP**" in the main menu of the touch screen.

3. Read-out of the gas flow at the reactor outlet, provided by a mass flow meter (MFM) situated on the gas outlet line (if this set-up option has been installed). This value can not be modified manually by the user.

4. **S1**: Additional control options for special configurations of the unit.

5. **S2**: Actuator for an additional loop control (opt). Both sensor and final control element (heaters) have to be connected to the "**AUX 2**" connector of the unit. Its P-I-D´s parameters and mode control (auto or manual) can be set in the "**MISC SETUP**" screen. If the unit incorporates the thermocouple for this option, the temperature loop control is shown over the "**S2**" button.

6. **S3**: Additional control options for special configurations of the unit.

7. **PELTIER**: Cooling / heating of the liquid-gas separator. The peltier configuration can be done in the "**PELTIER CONTROL**" menu of the touch screen, where the user selects the action (cooling / heating) and the desired output control for regulating the separator temperature. If this function is deactivated, the separator will be at ambient temperature. A display on the upper part of the tank shows the read-out for the liquid level in the liquid – gas separator, expressed as a percentage of the total volume of the tank (if the level sensor has been installed in the equipment). This value cannot be modified manually by the user.

8. **ALARM**: Consultation and deactivation of the system's alarm: When an alarm is triggered in the system, the icon "**Alarm**" will begin to flash, accompanied by a buzzer. By pressing on this icon, the alarm panel will be displayed (see Figure 4-7) where the cause of the alarm may be consulted (the icon that is flashing) and deactivated:

    - *RESET BUZZER* button, for deactivating the buzzer.
    - *RESET ALARM* button, for deactivating the alarm, provided that the situation of risk that triggered the alarm has been corrected in the system.

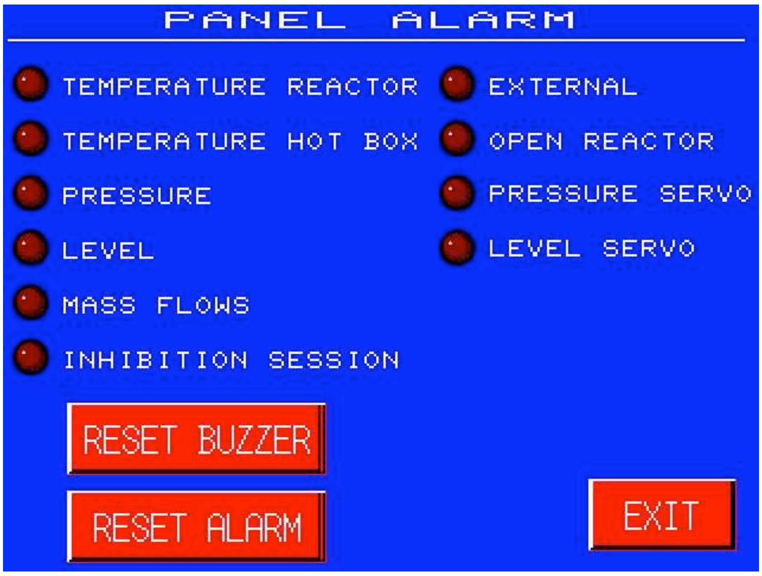


Figure 4-7

- o *Temperature Reactor*: The reactor temperature exceeds the maximum limit specified in its controller.
- o *Temperature Hot Box*: The hot box temperature exceeds the maximum limit specified in its controller. By default, this limit is set at 40ºC, with this being the temperature above which forced convection will be activated in the hot box. Accordingly, this alarm will normally be triggered, although this does not mean that the system is operating out of control.
- o *Pressure*: The pressure in the system exceeds the maximum limit specified in its controller.
- o *Level*: The level in the liquid - gas separator exceeds the maximum limit specified in its controller.
- o *Mass Flows*: Alarm for deviation of the flow of any one of the system's mass flow controllers regarding its set-point (consult the screen Mass Flow Set-up).
- o *Inhibition Session*: Whenever an alarm is triggered in the system, an inhibition session will be activated, interrupting the sequence of sessions programmed in the control software and so avoiding situations of risk in the system.
- o *External*: External alarm, additional to the reactor.
- o *Open Reactor*: Detection of open oven.
- o *Pressure Servo*: Alarm in the Digital Servo of the micrometric valve for pressure regulation in the system.
- o *Level Servo*: Alarm in the Digital Servo of the micrometric valve for regulating the liquid level in the liquid – gas separator.

Press the "**Exit**" key to return to the main menu.

9. **BYPASS**: Operating the reactor's by-pass valve: Access is by means of the "**Bypass**" key. When this icon is depressed, the valve is in by-pass mode, isolating the reactor.

10. **DOOR**: Opening / closing of the hot box door: It is operated by pressing the "**Door**" key.

4.5.2 MAIN MENU

Press the "**F1**" key on the touch menu to access the main menu, from where the following set-up screens can be accessed:

Figure 4-8

- *MASS FLOW SETUP*: Set-up menu for the mass flow controllers (MFC). The following is displayed for each one of the system's MFC's:
    - Maximum flow: These values are determined in accordance with the number of controllers of the equipment.
    - Units in which the gas flow is expressed.
    - Name of the gas.
    - % of alarm: Deviation alarm. This alarm is inhibited during the time specified in the *Delay Time Alarm* (in sec.), being triggered if the specified deviation persists during this time with respect to the set value

All these parameters may be modified by the user by pressing on their corresponding yellow boxes and entering the new values by means of the keys that are displayed on screen. Before beginning to work with the equipment, it is important to ensure that each reactant gas MFC installed has been properly set up, and that all the other MFC's not installed have their fields set to zero.

If the unit incorporates a mass flow meter (MFM), the user has to set its maximum flow in this screen, in ml/min. If the unit does not incorporate this option, this gap must be configured at 0 ml/min.

Figure 4-9

Press the "**Exit"** key to return to the main menu.

- *TEMPERATURE ALARMS*: Set-up menu for the temperature alarms.

This screen allows the user to select the actions the system is to carry out in the event of a temperature alarm. Pressing on the keys determines the following:

- **OFF:** Gases that will be shut down during the alarm (fuels, inflammables, reactants, etc.).
- **FREE:** Gases that will maintain the same status they had prior to the alarm: Inerts, for diluting the concentration of reactants inside the reactor).

Press the "**Exit"** key to return to the main menu.

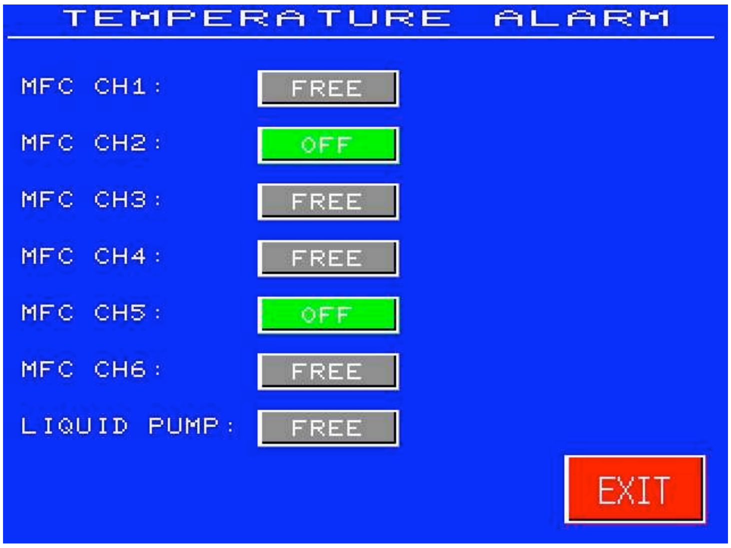


Figure 4-10

- *PRESSURE ALARMS*: Set-up menu for the pressure alarms.

Figure 4-11

As in the previous menu, this screen allows the user to select the actions the system is to carry out in the event of a pressure alarm. In this case, it is advisable to close off all gas and liquid inlets to the system in order to reduce the pressure within it (set them to **OFF**).

This alarm will remain locked and has to be reset manually on the alarm panel pressing the key “Reset Alarm”.

Press the “**Exit”** key to return to the main menu.

- *LEVEL SETUP*: Set-up screen for the level sensor (for equipment with this option).The description of this screen and the procedure for making the level sensor calibration is described in the section 4.6.3.3 of this manual.

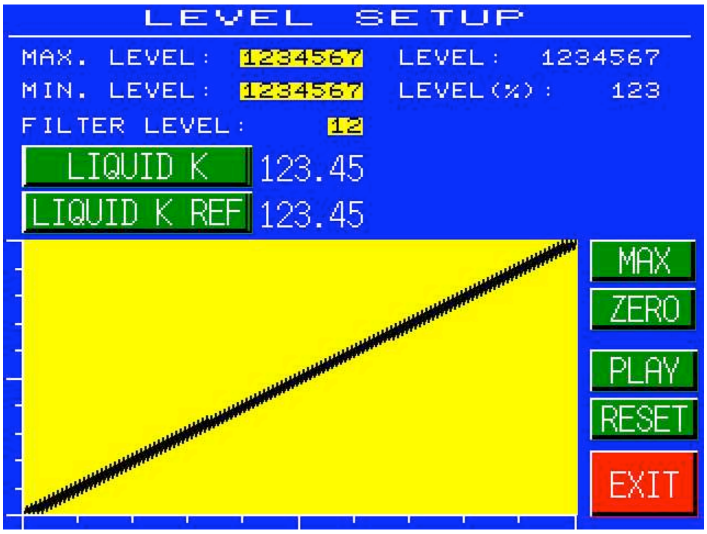


Figure 4-12

Press the “**Exit”** key to return to the main menu.

- *MISCELLANEOUS SETUP*: For setting up the following parameters:

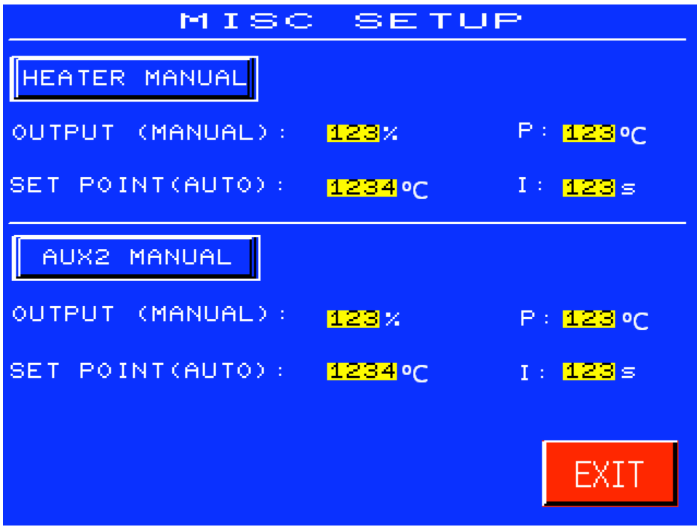


Figure 4-13

- HEATER: Heating of the system’s gas outlet line. The heater control can be set as:
    - “Heater Manual”: By default. In this mode, the user can set the output control (%) of the heater in the “Output (manual)”gap:
        - 0%: No heating
        - 100%: Maximum power of heating.

In this mode, the controller does no attend to the P-I parameters or the set point fixed by the user.

- o “Heater Auto”: For using this mode of control, the unit has to incorporate a thermocouple in the system’s gas outlet line. Working with this mode, the user can set the desired set point and the P-I parameters for the loop control (The controller does not attend the output of control).

***CAUTION**: If the unit does not incorporate this thermocouple, the selection of “Auto” mode of control inhibits the line heating (even when the button “**HEAT**” is activated in the main screen).*

- AUX2: Auxiliary control loop for temperature control (opt). If the unit includes this option, the heater must be plugged to the “**AUX2**” connector on the rear panel of the unit. The operation with this device is the same as has been described in the previous section “**HEAT**”.

Press the “**Exit”** key to return to the main menu.

- COMMUNICATIONS SETUP: Set-up parameters for the communications via Ethernet between the devices. By default, the unit is configured with the appropriated parameters to connecting it directly with the control PC with a crossed cable:

IP ADDRESS: 192.168.0.5

IP MASK: 255.255.255.0

GATEWAY: 192.168.0.1

TCP PORT: 1234

Figure 4-14

Press the “**Exit”** key to return to the main menu.

- PELTIER CONTROL: Selection of the peltier mode:
    - **COOL**: For reducing the L/G separator temperature (Minimum: about -1ºC)
    - **HEAT**: For increasing the L/G separator temperature (Maximum about 60ºC). *This mode is the recommended for working with high viscosity hydrocarbons, in order to avoid plugs in the liquid lines.*

- Peltier Control (%): This parameter is directly proportional to the cooling / heating power supplied to the L/G separator (values between 0 and 100 %)
- Duty time (s): Time of the cooling / heating cycle (typically 10 seconds).

Figura 4-15

- ADMIN SETUP: Screen for the system configuration by a PID Eng & Tech administrator. The password is not available for the user of the unit.

Figura 4-16

- ABOUT PID: Product information

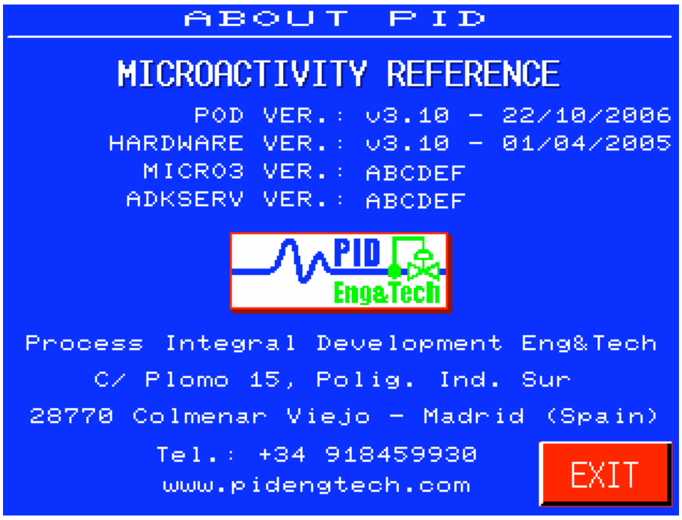


Figure 4-17

This screen provides information regarding the company PID Eng&Tech as well as the versions installed on the unit:

- Pod Ver.: Software for the touch screen
- Hardware Ver.: Software for the control panel
- Micro3 Ver.: Control software
- Adkserv Ver.: Embedded software

Press the “**Exit”** key to return to the main menu.

4.5.3 OTHER FUNCTIONS

- Adjustment of contrast on the touch screen: This function is accessed by means of the “**System**” key on the touch screen, and may be increased or decreased using the “**F3**” and “**F4**” keys.

4.6. SPECIAL CONFIGURATIONS OF THE MICROACTIVITY-REFERENCE

4.6.1 THE GILSON 307 HPLC PUMP

The Microactivity-Reference reactor provides the option of working liquids into the system. When this option is required, the system is fitted with a HPLC positive alternative displacement pump made by the firm GILSON.

4.6.1.1 Front view

1 – Digital screen

2 – Numerical keypad

3 – Pump head

4 – Pump head fastener

5 – Inlet to pump head

6 – Outlet from pump head

7 – Connections to manometer module

8 – Side attachment

9 – Side screws

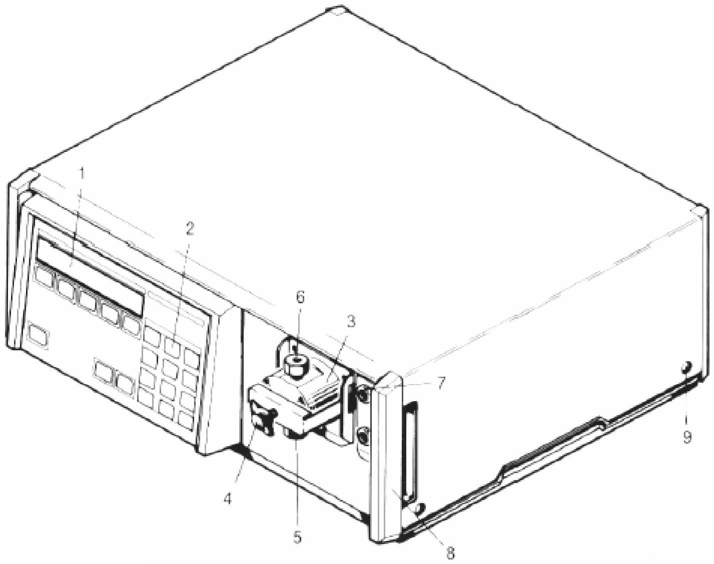


Figure 4-18

4.6.1.2 Keyboard

1 – Power indicator

2 - Screen

3 – Function keys: Their function is displayed on the screen and changes according to the menu.

4 - *PRIME*: The pump runs with maximum flow until *STOP* is pressed.

5 - *HELP*: It displays messages and instructions regarding the system, with no effect on pump operation.

6 - *CANCEL*: It clears the latest entry without storing it in the memory.

7 - *ENTER*: Confirmation key

8 – Numerical keypad.

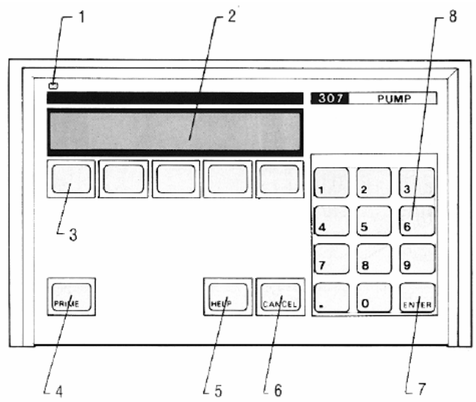


Figure 4-19

4.6.1.3 Technical Specifications

<table>
<tr><td>Pump</td><td>Programmable reciprocating pump with single-piston interchangeable head, constant stroke and fast refill motion, internal pulse dampener and pressure feedback</td></tr>
<tr><td>Pump Heads

SC = stainless steel
W = rinsing compartment for aqueous salt solutions (≥0.1M)
Ti = titanium</td><td><table>
<tr><th rowspan="2">Pump Head Model</th><th rowspan="2">Flow Rate Range (mL/min)</th><th colspan="2">Pressure Range</th></tr>
<tr><th>psi</th><th>bar</th></tr>
<tr><td>5SC</td><td>0.010–5</td><td>14.5–8700</td><td>1–600</td></tr>
<tr><td>10WSC</td><td>0.050–10</td><td>14.5–8700</td><td>1–600</td></tr>
<tr><td>10WTi</td><td>0.050–10</td><td>14.5–8700</td><td>1–600</td></tr>
</table></td></tr>
<tr><td>Mixing Modules</td><td>811D Analytical Mixer: 1.0–10 mL/min, 1.5 mL; 110/220V
811D Titanium Analytical Mixer: 1.0–10 mL/min, 1.5 mL; 110/220V
811D Titanium Analytical Mixer: 0.1–3.0 mL/min, 0.7 mL; 110/220V
811D Microbore Mixer: <0.1 mL/min, 65 μL; 110/220V</td></tr>
<tr><td>Operating Modes</td><td>Constant flow rate (Flow), constant volume (Dispense) and time-based sequence (Program)</td></tr>
<tr><td>Pump Parameters</td><td>Head number: 5 or 10
Liquid compressibility: 0–2000 $Mbar^{-1}$
Refill time: 125–1000 ms
Inlet pressure: 0–10 MPa</td></tr>
<tr><td>Programmable Parameters</td><td>• Timed events for programming four output contact closures and one input to wait for injection
• Time, adjustable from 10^{-2}–10^{4} min with increments from 0.01–1 min depending on the range used
• Flow control adjustable in mL/min from 0.01%–100% of the maximum flow rate of the pump head being used
• Up to 999 loops with unlimited linking of files
• Storage for 10 user programs and four error files with a maximum of 25 points and timed events in each program</td></tr>
<tr><td>Flow Rate</td><td>Flow rate: 10 μL/min–10 mL/min
Coefficient of variation: 0.1%–0.6% with aqueous solutions or hydro organic polar solvent mixtures; 0.3%–1% with hydrocarbons or chlorinated volatile solvents
Maximum accuracy error: ±1% with water over the full flow rate and pressure ranges</td></tr>
<tr><td>Pressure</td><td>0.1–60 MPa (600 bar, 8700 psi), depending on pump head used
Accuracy: <±1% or 0.1 MPa (1 bar, 15 psi)
Repeatability: <1% or 0.1 MPa (1 bar, 15 psi)</td></tr>
<tr><td>Pulse Dampening</td><td>Pulsation: <1% with water at 1 mL/min and pressure >8 MPa
Dampener volume: 0.6 mL at atmospheric pressure, 1.6 mL at 60 MPa</td></tr>
<tr><td>Software</td><td>Via Gilson UniPoint® System Software</td></tr>
<tr><td>Communication Interface</td><td>RS-232 or GSIOC; four inputs and four relay outputs</td></tr>
<tr><td>Display Panel</td><td>2 x 24-character LCD</td></tr>
<tr><td>Front Panel</td><td>Keypad and built-in help messages</td></tr>
<tr><td>Liquid Contact Materials</td><td>316L stainless steel, titanium, sapphire, ruby, PTFE, PCTFE and HDPE</td></tr>
<tr><td>Power Requirements</td><td>Frequency: 50–60 Hz
Voltage: 100–120 or 220–240V; mains voltage fluctuations not to exceed ±10% of the nominal voltage</td></tr>
<tr><td>Environmental Operating Temperature</td><td>0–40°C</td></tr>
<tr><td>Manufacturing Standards</td><td>Meets applicable Safety and EMC certification standards; CE certified</td></tr>
<tr><td>Instrument Dimensions (w x d x h)</td><td>33 x 33 x 15 cm (13 x 13 x 6 in.)</td></tr>
<tr><td>Instrument Weight (with head)</td><td>11.5 kg (25.4 lbs.)</td></tr>
<tr><td>Shipping Weight (with head)</td><td>15.4 kg (34 lbs.)</td></tr>
</table>

4.6.1.4 Switching on the pump

Before operating the liquids pump, the electrical and mechanical installation of the system needs to be performed, which means it is advisable to consult the pump's user handbook.

When the pump is switched on (the switch is to be found on the rear), the screen displays a message indicating the model of pump as well as the version of the control software, as shown below.

```
Pump Model 307 V x.xx
```

Following this message, the pump's main operating screen is displayed.

4.6.1.5 Setting up the pump

Once the electrical installation is done (as is described in the chapter 3.2.3 of this manual), it is necessary to configure the pump (if the MA-Ref reactor includes the pump, it is already configured at PID Eng & Tech laboratories, so it is no necessary for the customer to do it again):

- Setup pump hardware (PUMP): Press "Menu" and "Pump". The sequence of parameters is:

- ***Refill time:*** It is the time required for the piston return stroke. Normally it is set at the lowest value (125 ms). If cavitation of degassing occurs, then a higher value must be used. The minimum value is 125 ms and the maximum value is 1000 ms.

  The maximum flow rate depends on the refill time. If the refill time is too long, a message “Invalid settings” flashes when you run the program. The refill time or flow rate must be lowered.

- ***Pump Compressibility***: This data is used to calculate the flow rate compensation for the compressibility of the solvent. The minimum value is 0 and the maximum is 2000 $Mbar^{-1}$. The default value is 46 (compressibility value for the water). Compressibility values for the common solvents at atmospheric pressure are listed:

Solvent	***Xo (Mbar-1)***
Carbon Dioxide	1150
Water	46
Methanol	123
Acetonitrile	99

- ***Pump Head Size***: This parameter is the size of the pump head. Possible values are 5, 10, 25, 50, 100 and 200. It is possible to use any head size with the Gilson 307 pump. However, to ensure accuracy, reproducibility and efficient pulse dampening, the flow rate should not exceed 5 ml/min.

- ***Inlet pressure***: This is the pressure at the inlet of the pump head. This allows the accurate pumping of liquefied gas. If must be set to the same as the pressure of the aspirated liquid, that is the saturating vapour pressure at the ambient temperature for the liquefied gas delivered from a pressurized cylinder. When using carbon dioxide at a temperature of 22ºC, the value of the inlet pressure should be defined as 6 MPa. A table of inlet pressures is shown below:

Ambient temperature (ºC)	15	20	22	25	30	31	(Tc)
Pressure Po (Mpa)	5.1	5.8	6.0	6.5	7.2	7.4	(Pc)

  The default value is 0 Mpa.

- Input / Output parameter setup (I/O): Press “Menu” and “I/O”. The sequence of parameters is:

- ***High pressure limit:*** If the pressure reading from the manometric module rises above this limit, the pump will stop. The sequence following a high pressure error is described later in this chapter. The pressure can be displayed in three different units, bar, MPa or kpsi. Change the units by pressing the soft key below the units display, bar, MPa or kpsi. The maximum value is 600 bars. The default value is 600.

- ***Low pressure limit***: If the pressure reading from the manometric module drops below this limit, the pump will stop. The minimum value is 0. Default value is 0.

- ***Alarm***: The alarm is a buzzer which sounds every time there is an error or an invalid setting. It can be programmed to be either On or Off. This function only controls the operation of the buzzer, it does not affect the operation of the pump when there is an error. If the alarm is On, the warning buzzer will sound every time there is an error. An error can be a pressure limit, or an invalid setting. This parameter can be changed from On to Off and vice versa by pressing the soft key Change.

- ***GSIOC Unit identification number***: A Gilson system can be controlled from a computer using a GSIOC interface and GSIOC cables. Each instrument in a system must have a unique identification number to distinguish it from other equipment connected to the GSIOC communications channel. The GSIOC identification number in the 307 can be set between 0 and 63.
  The default value is 1.

- ***Output XX is open/Closed:*** There are four relay outputs in the 307 pump numbered 1, 2, 3 and 4. These outputs are used to control other instruments. They can be programmed to open and close during a method run. They can also be opened and closed manually.
  The default state is open.

- ***Zero pressure reading***: The Zero soft key is used to set the pressure reading to zero when there is zero pressure in the system. This ensures accurate pressure readings when the pump is running. Before pressing Zero, make sure that the pump has stopped and the pressure has dropped to zero, otherwise further pressure indications will be incorrect. If the operation is successful, the message 'Pressure reading is zero' is displayed. If the operation is not successful due to pressure in the system, the message 'Not done, check pressure' is displayed.

4.6.1.6 Venting the pump

The system needs to be vented before the liquids pump is operated. This involves filling the inlet tank with the liquid that is to be introduced and following these steps:

- Adjust the syringe on the upper part of the 3-port valve.
- With the valve in the position "load syringe" (Figure 4-20, position no. 1), draw on the syringe's plunger, thereby introducing liquid inside, removing air bubbles from the feed line.
- Set the valve to the position "Injection from syringe" (Figure 4-20, position no. 2) and press the "**Prime**" key on the keyboard. The pump will then begin to pump liquid at its maximum speed. Press the plunger on the syringe until no air bubbles can be seen at the pump inlet and liquid reaches the outlet.
- Set the valve to its normal operating position (Figure 4-20, position no. 3). When no more air bubbles are observed at the outlet on the liquids pipe, press the "**Stop**" function key to finish venting the pump.
- Under normal operation, plugging the end of the feed line will increase the pressure displayed on the message screen whilst the line is sealed and the pump is running.

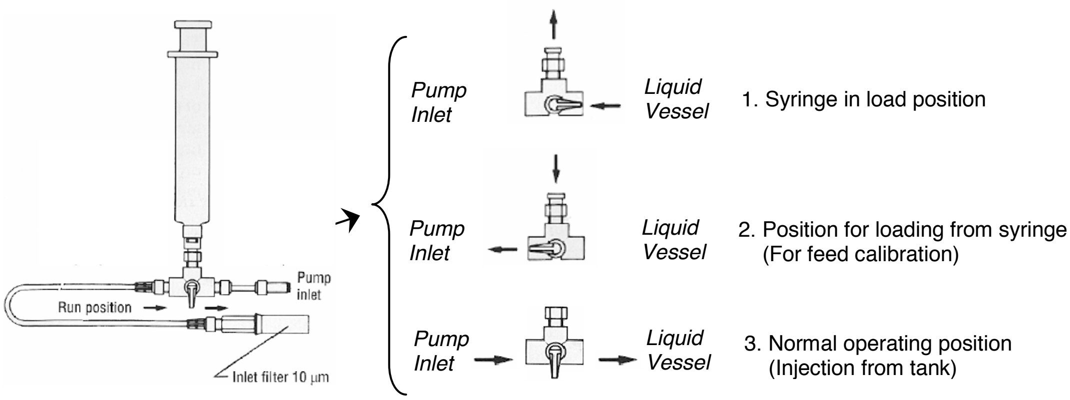


Figure 4-20

4.6.1.7 Pump operation

After entering the data about the pumping system, the pump is ready to run. The 307 pump can operate in 3 different modes. These modes are:

- **Flow**: The 307 pump provides a constant flow rate. The pump starts when the “**Run**” key is pressed and stops when the “**Stop**” key is pressed.

- **Dispense**: The 307 dispenses a specified volume. The pump starts when the “**Start”** key is pressed and stops when the specified volume has been dispensed.

- **Program**: The 307 controls a complete system. In this mode, the 307 pump can create gradients of flow rate, open and close outputs to control other instruments and wait for signals from other instruments.

By default, the pump is programmed to operate in **Flow** mode, meaning that it will always provide a constant flow, determined beforehand by the user. This means that operating the pump is perfectly straightforward:

1- Enter the desired flow in ml/min using the numerical keypad and press “**Enter**”. Any flowrate may be set that ranges between 0.01% and 100% of the size of the pump head (5SC head = 5 ml/min). If the flow introduced exceeds this value, the message “**Invalid settings**” is displayed on the screen after pressing “**Run**”. In such a case, the value has to be modified. During pump operation, the flow may be modified as often as required without having to stop it.
2- Press “**Run**” to start up the pump.
3- Press “**Stop**” to stop the pump.

This is the normal operating mode. For further information regarding all the other operating modes, as well as the different set-up options for the pump and troubleshooting, consult the Gilson 307 pump’s handbook.

Attention!!

It should be noted that once the system is controlled by the Process@ acquisition software and, therefore, the pump operates on a remote basis by means of digital communications, control of the pump ceases to be manual, as has been described up until now, and all modifications in its operating mode are to be made through the control PC. If the need arises to use the pump’s keyboard and functions, it will be necessary to reboot the pump.

When the pump is going to work at atmospheric pressure (outlet pressure = atm.), it is necessary to install at the liquid outlet a Back Pressure Regulator, that generates a outlet pressure of 34- 35 bar.

4.6.2 PRESSURE CONTROL

4.6.2.1 Introduction to pressure control systems in microactivity reactors

The use of a reactor, such as the Microactivity-Reference, for catalytic microactivity studies involves the use of extremely low streams and, usually, high pressures. This implies extremely low values for the flow or stream coefficient (Cv) that characterises the regulating valve, of around 10^{-7} to 10^{-4} (with this coefficient being defined as the flow of water in US gallons per minute that passes through a valve in a fully open position and with a loss of load of 1 psi).

To ensure high accuracy in the pressure control of the system in the Microactivity-Reference unit, as well as a non-pulse gas flow inside the reactor, several different alternatives that are used commercially in systems like the following:

- Tescom type backpressure: Control is only proportional, which means it is affected by an offset error. These systems are not suitable for microflow systems and they present high dead volumes. They generate a pulse gas-flow through the reactor.

- Electronic back-pressure, with MFC type valve: Despite providing an extraordinarily stable gas-flow in the system's pressure control, its main drawback appears when the system contains products in the vapour phase, when microdroplets of condensate may accumulate on the control valve's orifice. These systems do not allow for heating, given their electronic nature.

- Control loop with control valve: The problem of control in previous systems was resolved by configuring a control loop for pressure, whereby the signal from a pressure transmitter is received by a PID controller that produces a control signal that is relayed to a control valve that acts upon the system's output current, modulating the circulation flow and thus regulating the pressure.
  The problem posed by this control system is the availability of commercial microvalves that are suitable for microflow systems, basically consisting of a cylindrical orifice through which a round rod is inserted whose purpose is to vary the fluid's length of passage through the microvalve. These systems feature very low rangeability, of around 10 (difference of flow that passes through the valve between the minimum and maximum aperture for a specific pressure), whereby they are not suitable for a system such as the Microactivity-Reference, in which widely differing operating conditions are to be studied and whose regulating valve should have rangeabilities of around 160.

4.6.2.2 The micrometric regulating valve

The Microactivity-Reference includes a micrometric regulating valve for pressure control in the reactor (as well as for level control in the liquid – gas separator in those systems that include this option) of very high rangeability, consisting of a needle whose displacement creates an increasingly tighter fit within an orifice, generating a variable section passage that depends on the distance the needle has been moved (Figure 4-21).

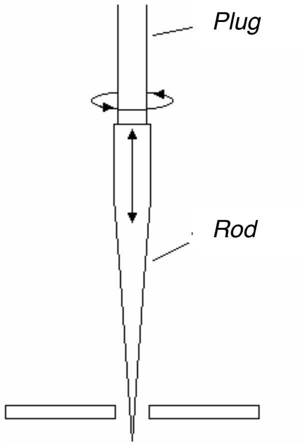


Figure 4-21

This kind of valve furthermore improves rangeability, control precision, as displacement is not performed linearly, but rather by means of the turning action of the rod caused by a micrometric screw. If the shaft of the screw allows for 10 turns from the fully open position through to the fully closed position and each turn is a full 360°, fitted with an actuation system such as has been designed for this unit and which distinguishes the position with an accuracy of 1 degree of circumference, there is a total of 3600 possible states for the relative orifice/needle position, which means a precision for the system's pressure control of ± 0.1 bar without permitting sudden variations in the gas-flow at the reactor outlet of more than 5% of the total flow passing through the reactor bed.

In a study carried out with 8 commercial micrometric regulating valves, the one providing the best results in the Microactivity-Reference reactor operating with flows below 50 ml/min and pressures higher than 50 bar is one made by Hoke, model 1315G2Y, which has the following specifications:

- Maximum operating pressure: 345 bar at 21ºC.
- Range of operating temperatures: from –54ºC to 232ºC (it may be found fitted inside the hot box, so avoiding the formation of condensates in the orifice).
- Dyna-Pak gasket, which ensures the tight sealing of the rod without major compression on the same.
- Construction material: 316 Stainless steel.
- Dead volume: ≤ 0.2 ml.
- Connections 1/8´´.
- Original orifice: Replaceable, in 316 stainless steel. The need to operate in the proximity of the close contact between the orifice and the needle inevitably leads to wear on the needle caused by rubbing. Consequently, the disc containing this orifice on these valves (5 mm diameter, 1.8 mm thickness and orifice 1.19 mm) has been replaced by a replica made of PEEK (polyetheretherketone), a chemically inert material that has an excellent mechanical performance, withstanding high operating temperatures and featuring self-lubricating properties with major hardness and resistance to distortion. Once it has been distorted by continuous use of the valve, this disc may be replaced as often as necessary.
- The manufacturer's original C_V curve may be seen in Figure 4-22. Modification of the orifice on the valve and its replacement with another made of PEEK alters its C_V curve near to the closed position. Experimentally, an excellent regulating performance is achieved in C_V scenarios of 10^{-5}, that is, the modified valve accurately regulates flows of even 40 or 20 ml/min with pressures of 60 to 90 bar.

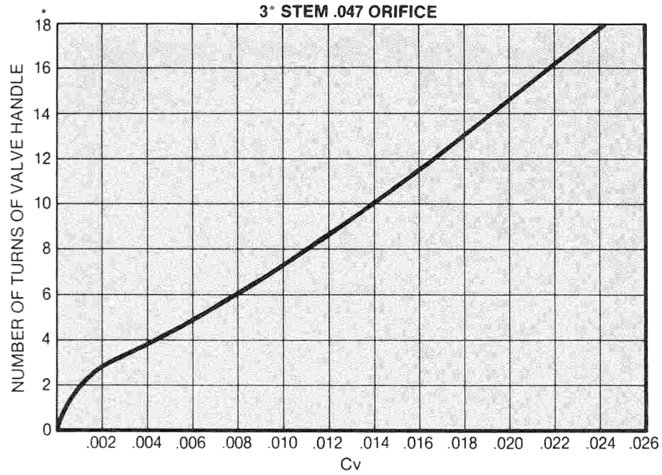


Figure 4-22

The system that has been designed for operating on the valve consists of a high-resolution microstep motor. Such motors consist of electromagnets that are connected and disconnected alternately so that a rotor (permanent magnet) moves in small steps in the required direction. By multiplying the number of coils on the motor or creating complex systems for the switching of the coils regulated by a microprocessor, use can be made with stepping motors of 200 steps per turn in set-ups with 51,200 steps in one rotation.

The motor's shaft is coupled to the micrometric valve and to a potentiometer, allowing for the position of the valve with 1^{st} resolution to be read at any given moment. The position's control system operates on a digital basis by successive approximation, comparing the present position (relayed by the potentiometer) to that specified by the order, then calculating the number of positions that remain to be advanced and all within an infinite loop that constantly corrects the motor's position with the system's extremely rapid response time. To improve the potentiometer's reading of the position, it has been used a current generator and an instrumentation amplifier, eliminating errors and noise stemming from the cabling.

The pressure control valve is located downstream of the reactor, once the reaction gases have passed through the separator / condenser.

The following are the characteristics of the pressure control system:

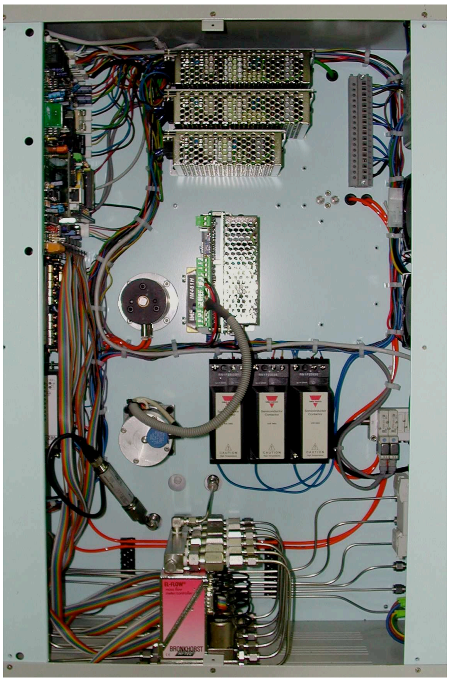
- Range of operating pressures: atmospheric – 100 bar.
- Control accuracy: ± 0.1 bar.
- Variations in gas-flow at the reactor outlet ≤ 5%.
- Maximum heating temperature of the valve: 200ºC (the valve is inside the hot box: see Figure 2-4).

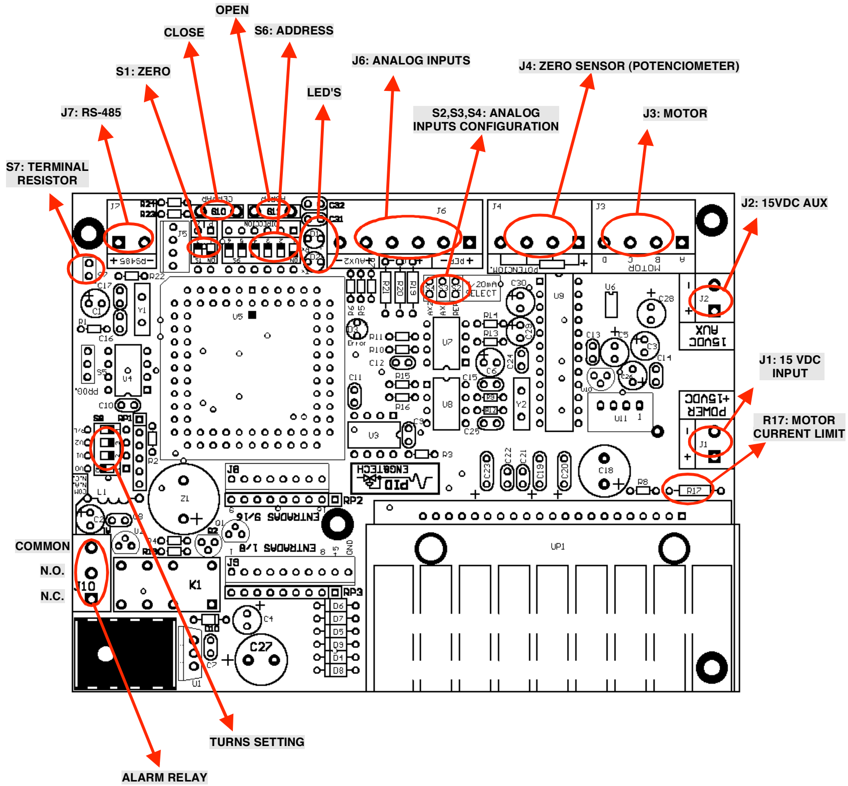
Pressure control is carried out by means of the TOHO TTM-005 controller that is to be found on the front of the reactor (see Figure 2-3), and its operation is described in section 4.2 of this manual.

4.6.2.3 Configuration of the Servo Digital V4.0 unit (microstep positioner)

The Servo Digital V4.0 microstep positioner that is part of the pressure and level control systems on the Microactivity-Reference units are factory set with the optimum parameters for the equipment’s correct operation, which means that, in principle, the end user should not need to configure this unit.

In case that the customer needs to modify these parameters, here we include a detailed description of the printed circuit in the microstep servo digital unit. Access to it involves unscrewing the metal plate on the right-hand side of the reactor, as shown in Figure 4-23:





Servo Digital V4.0 unit for the positioning of the micrometric valve for pressure regulation.

In those units that include the level control system for liquids in the condenser, their corresponding Servo Digital unit is to be found to the right of this system.

Figure 4-23

- **Zero calibration (S1):**

The valve’s “zero” setting, or the point at which it is fully closed, is factory set, but frequent and constant use of the equipment may alter that point as a result of minor distortion of the PEEK disc that contains the valve orifice. This effect is verified as follows:

- Set the pressure and level controllers to manual mode and fully close the valve (0%). Bring the system up to operating pressure. Submerge the system’s gas outlet into a water-filled container and check that there is no bubbling. If gas is observed to be escaping, the values of the valve’s zero point will have to be lowered (more closed).

To adjust the valve's zero-setting, set switch "**Z**" on the dip-switch to "**On**". (S1: zero, see Figure 4-24). The motor is now free for manual positioning (by means of the corresponding TOHO controller in manual mode, acting on the % of control output) or by pressing the microswitches "**Open**" or "**Close**" the valve's new zero-setting (point at which the system's gas outlet ceases to bubble). If the buttons "**Open**" or "**Close**" are pressed, the motor moves the shaft in 5º jumps. Once this has been performed, return the "**Z**" switch to the "**Off**" position. The new zero-setting will be saved on memory.

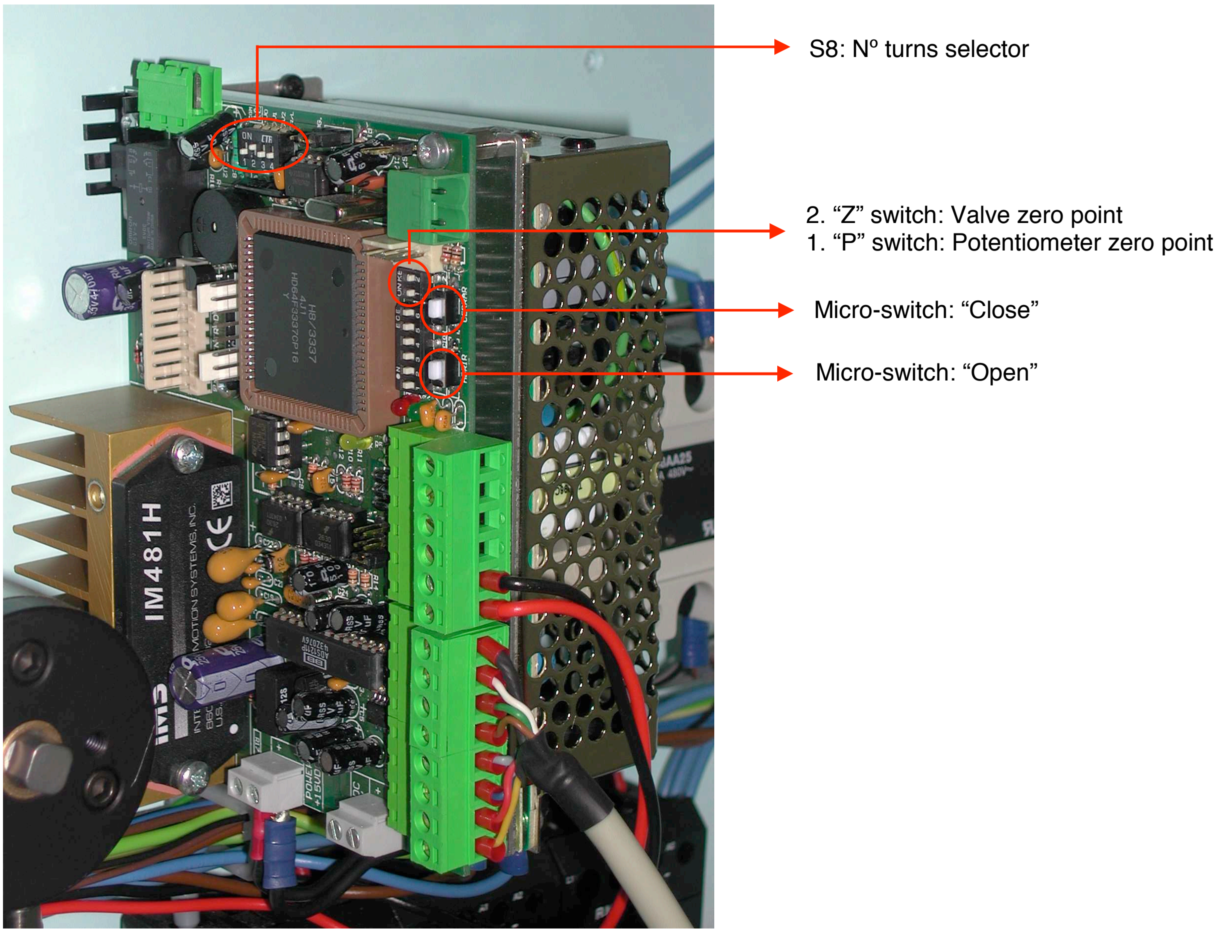


Figure 4-24

To return to the initial default zero-setting (2nd turn of the potentiometer) turn switch "**P**" on the dip-switch S1 to "**On**" and then to "**Off**". The new zero will be set on the 2nd turn of the potentiometer.

- Turn off the unit
- Turn on the unit again
- Once the previous step has been completed, open the valve 5% and check that the there are bubbles at the gas outlet. If there are not, the values for the valve's zero setting will have to be raised (more open).
- To set the zero point for the liquid valve, the user has to follow the same procedure as the described before.

- **Selecting the number of turns (S8):**

This parameter represents the number of turns that the valve is going to do from the 0% to the 100 % of output control (from “totally closed” to “totally opened”).

By default, the pressure and level control valves on the Microactivity-Reference are set with the number of turns that ensures optimum performance under the unit’s normal operating conditions.

However, if the user changes the operation conditions (modifying the total gas flow), it would be necessary to change the maximum number of turns:

- If the gas flow increases, and the valve have to open the 70-75 % for controlling the desired pressure, it would be advisable to increase the number of turns.

- If the gas flow decreases and the valve opens just 5-15 % for controlling the desired pressure, it would be advisable to decrease the number of turns.

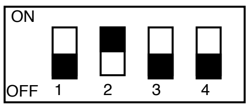
It is recommended that the valve opens from the 20 to the 65 % for controlling the desired pressure.

For changing the number of turns, proceed as following:

- Localize the dip-switch S8 of the driver (see Figure 4-24)

- Check that the switch 4 on dip-switch S8 is on the “**Off**” position (see Figure 4-24)

- The number of turns is determined with switches 1, 2 on 3 on dip-switch S8:



No. OF TURNS	SWITCH POSITION (S8)		
	1	2	3
1	off	off	off
2	on	off	off
3	off	on	off
4	on	on	off
5	off	off	on
6	on	off	on
7	off	on	on
8	on	on	on

- Turn off the unit

- With the help of a screwdriver, change the position of the switches 1, 2 and 3, according with the table above.

- Turn on the unit again

- Now, the valve is configured with the new number of turns.

4.6.3 LEVEL CONTROL IN THE LIQUID – GAS SEPARATOR

4.6.3.1 Introduction to level control in microactivity reactors

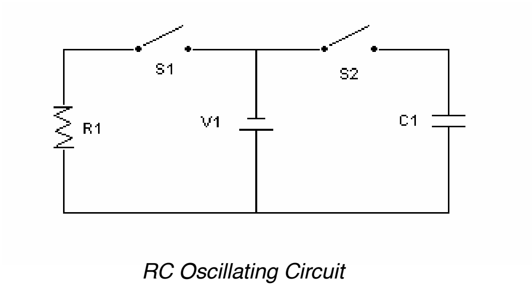
In those systems in which the aim is to monitor the reaction in real time, continuous collection has to be made of the condenser liquid in the liquid – gas separator for its subsequent analysis.

In a reactor operating at atmospheric pressure, the removal of condensed liquids in the separator has to be performed manually by an operator. But this procedure is not possible in equipment that is operating at a pressure that is higher than the atmospheric one, as the loss of the hydraulic seal on the liquid products at the bottom of the separator would lead to a major leakage of gasses into the atmosphere, and possibly cause an accident. In this case, the liquid-gas separator is to have a control loop whereby, as of the recording of a specific level of liquid, a control valve is operated to maintain the liquid level constant, or what is tantamount to the same thing, continuously remove each new drop that is formed in the separator.

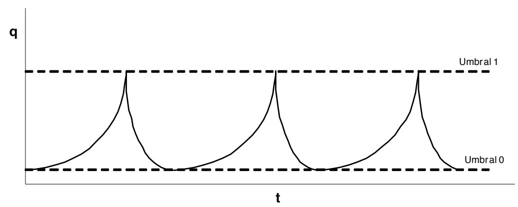
Other commercial systems applied in pilot plant situations base this level reading on systems that record the differential pressure between the ends of the separator. Thus, the pressure at the base of the separator is that corresponding to the pressure in the installation plus the pressure corresponding to the height of the liquid column present in the separator. Yet this technique presents serious problems when it is used in the measurement of a microvolume: the errors inherent to this technique when measuring the level of a tank that collects the condensates in a system that increases at the rate of, for example, 0.05 ml/min, renders this technique unviable for use in a reactor for studying catalytic microactivity.

4.6.3.2 The capacitive level sensor

With a view to resolving the problems posed in systems of this kind, a liquid-gas separator has been designed with a capacitive type level sensor with a very low dead volume. With this system, when liquid is present between the isolated probe and the chassis on a metal tank, this liquid behaves as a dielectric, altering the electrical capacity of a condenser system. An RC oscillating circuit, such as the one shown in Figure 4-25, will then provide a frequency signal proportional to the system's capacity and which is, therefore, proportional to the amount of liquid in the tank. Without considering geometric issues in the design of the tank, this circuit's output signal will be directly proportional to the height of the liquid in the tank.



RC Oscillating Circuit



Oscillating Circuit behaviour

Figure 4-25

This system's output signal is also proportional to the dielectric constant of the substance that acts as dielectric. Accordingly, the greater the difference of dielectric constant between the process liquid and the air (or gas that occupies the space not taken up by the liquid) the greater the output signal the system will generate.

The capacitive level sensor used is inserted through the upper part of the liquid – gas separator described in section 4.3 of this manual, and consists of a 3 mm diameter probe that is electrically isolated from the rest of the system by means of elastomer-type seals, chemically compatible and withstanding pressures of up to 400 bar.

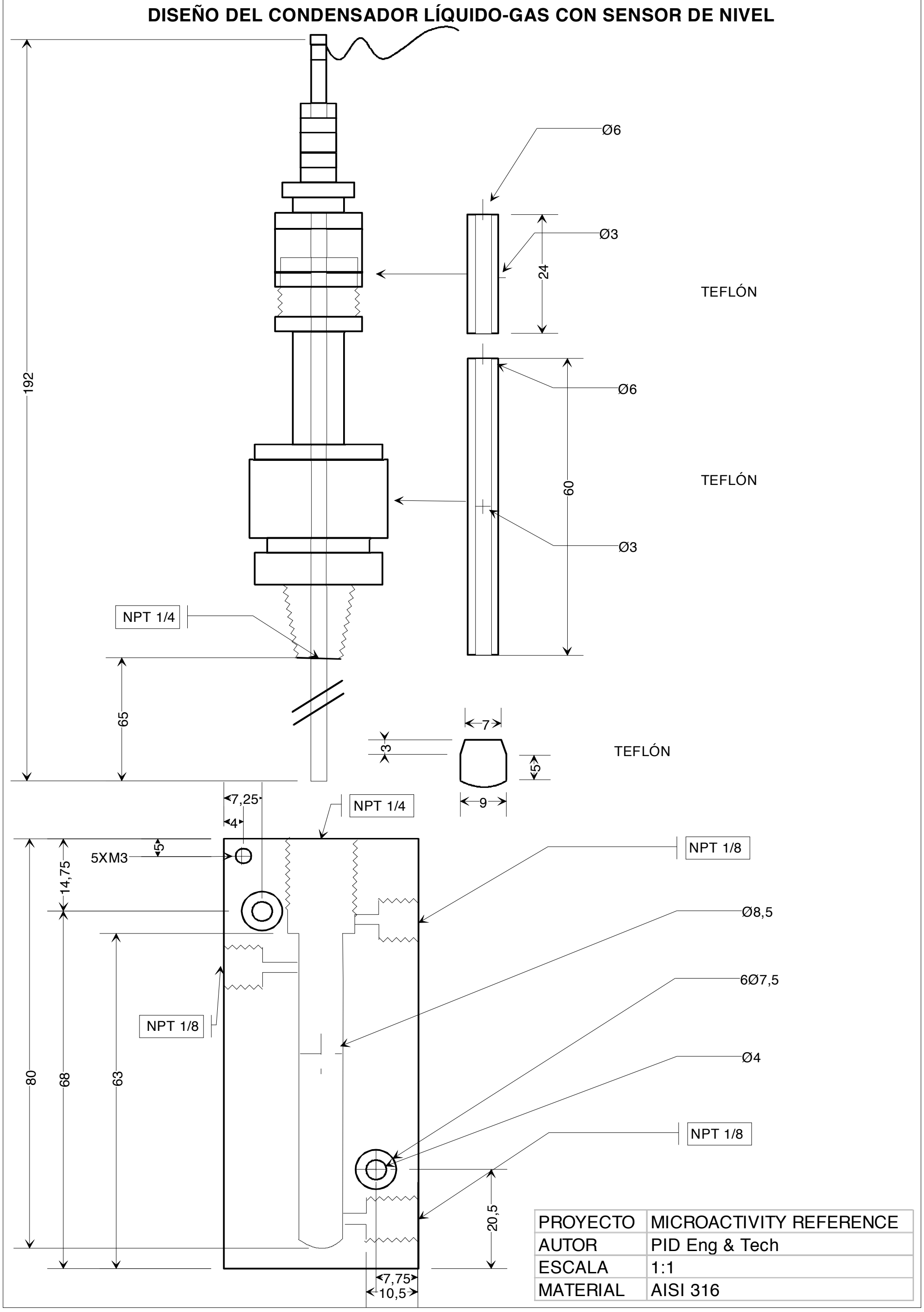
The design of the liquid – gas separator with level sensor incorporated is featured in Figure 4-26, where the parallelepiped piece may be observed, as well as the electrical insulator and the probe that, electrically insulated in this tank, becomes the condenser's second plate.

The photograph of the assembly (Figure 4-27) provides a detailed view of the piece that constitutes the system's electrical insulator and other parts, in Teflon, which besides guiding the assembly, serve to eliminate the system's dead volumes.

The separator – level sensor assembly is connected to a micrometric regulating valve that is connected to a microstep motor, like the one used in the system's pressure control (see section 4.6.2 of this manual), which means that recording the level inside the tank and operating said valve allow for the removal and collection of liquids on an automatic and continuous basis in real time, with a control accuracy of ± 0.01 ml.

As in the case of the system's pressure control system, the Servo Digital V4.0 microstep positioner unit that includes the level control system in the separator on Microactivity-Reference units is factory set with the optimum parameters for the correct operation of the equipment, which means that the end user should not, in principle, configure this unit. However, frequent and continued use of the sensor may in time make it necessary to carry out the zero calibration of the valve or select the number of turns of the same. In these cases, the user is to proceed in the same way as outlined in section 4.6.2.3 of this manual, but acting on the V4.0 servo digital unit corresponding to the level control valve, which is accessed by unscrewing the metal plate on the right-hand side of the reactor.

Level control in the separator is carried out by means of the TOHO TTM-005 controller that is located on the front of the reactor (see Figure 2-3), whose operation is described in section 4.2 of this manual.



PROYECTO	MICROACTIVITY REFERENCE
AUTOR	PID Eng & Tech
ESCALA	1:1
MATERIAL	AISI 316

Figure 4-26

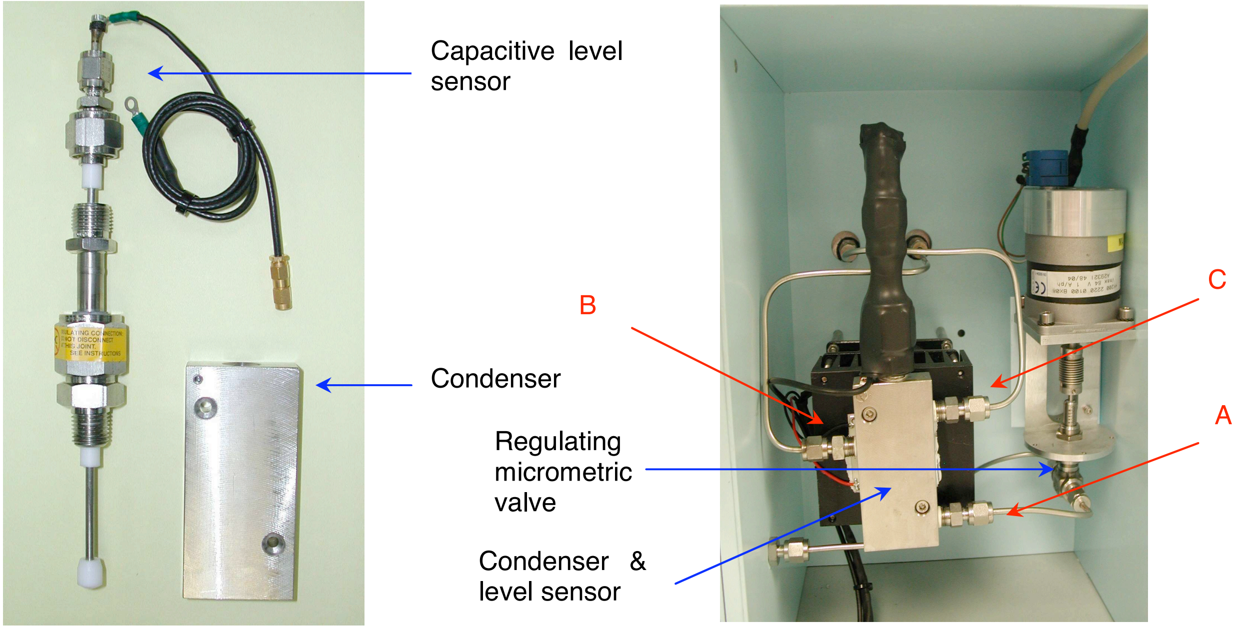


Figure 4-27

4.6.3.3 Calibrating the level sensor

All Microactivity-Reference equipment that includes the level control option in the liquid – gas separator is factory supplied with the level sensor calibrated with distilled water, which means that the user should not calibrate it again when beginning to operate with the equipment. In this state, the TOHO level controller that is on the equipment's front panel will display the reading of the ml of water inside the separator at any given moment.

There is no need to calibrate the level sensor on a regular basis, although it is advisable to perform a calibration whenever the user dismantles any part of the liquid – gas separator, as well as whenever any anomalies are detected in the reading of the level inside. In such cases, proceed as follows:

- Switch off the Microactivity-Reference unit.
- Loosen and remove the 3 connectors on the tank that are shown in Figure 4-28 in the following manner:

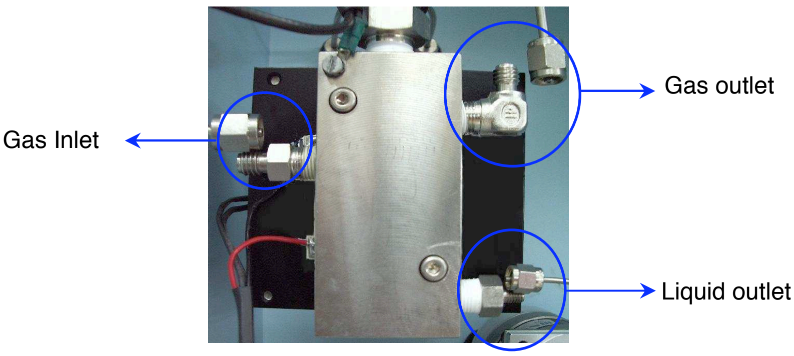


Figure 4-28

- o Use a 5/16 spanner to loosen and remove the liquid outlet connection on the tank.
- o
- o Use a 7/16 spanner to loosen the gas inlet and outlet connections.

- Place a container below the liquid outlet.

- Clean and dry the tank to remove any possible dirt. Cleaning may be performed by applying Helium, compressed air, etc.

- Place a 1/16 plug in the liquid outlet.

- Switch on the Microactivity-Reference unit.

- For a graphic display of the liquid level in the tank during calibration, press the key "**F1**", **"Level Setup"** and **"Play"** on the touch screen.

-

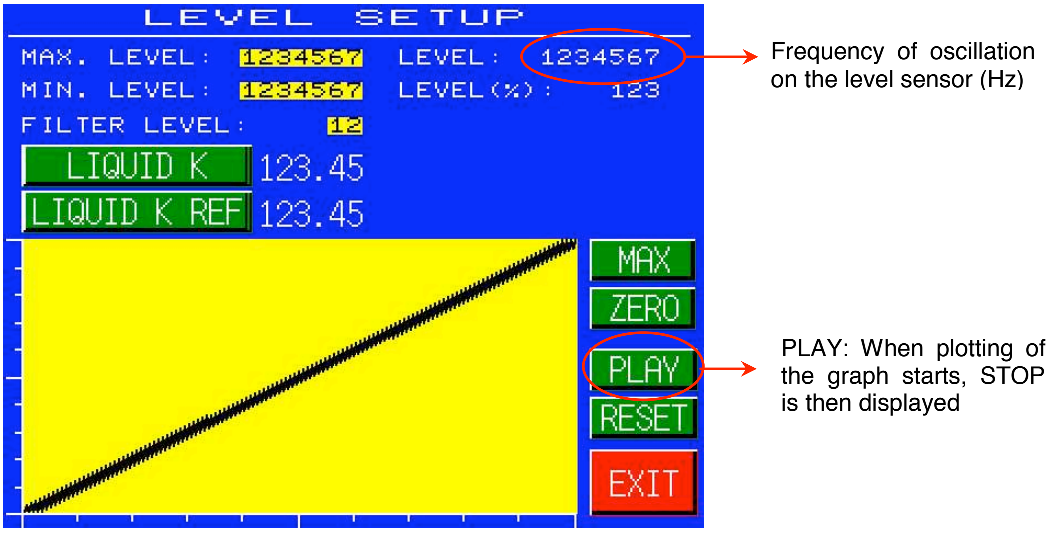


Figure 4-29

- *Calibrating the sensor with deionised water* (dielectric constant = 80.1):

  - Select the reference liquid (liquid used to calibrate the device): press the button "LIQUID K REF" and select the liquid in the list represented in Figure 4-30. If the reference liquid does not appear on the list, select the option "OTHER" and introduce its dielectric constant in the yellow background field.

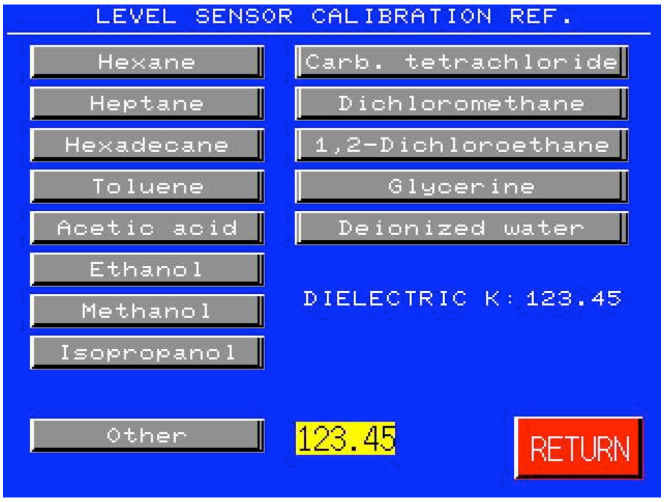


Figure 4-30

Press the "**RETURN**" key to return to the level sensor calibration screen.

  - Select the condensed liquid that, in this case, is the liquid used in the calibration (reference liquid). Proceed as described before: press the "**LIQUID K**" button and select the liquid.

Once the level sensor is calibrated and the equipment making reaction, the user should select the reaction product liquid in this screen. If this is unknown or it is a mixture, the user can select the option "**OTHER**" and estimate the mixture dielectric constant.

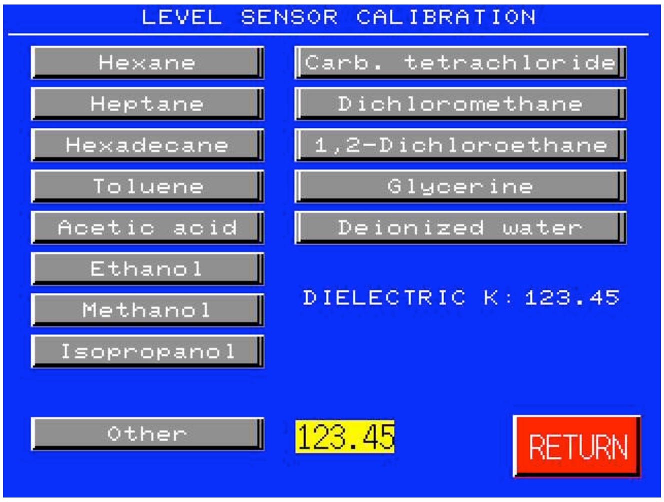


Figure 4-31

- Prior to starting calibration, observe that the oscillation frequency registered on the screen in the field "**Level**" is practically constant. This value corresponds to the sensor's baseline (volume of liquid in the tank = 0), and can be saved on memory pressing the button "**MIN**" in the touch screen: the value is saved on the field "**MIN.LEVEL**".

- Use a graded syringe to insert 0.5 ml of deionised water through the liquid inlet orifice, keeping the liquid outlet sealed with the plug (insert the needle right inside the tank to avoid the meniscus effect at the inlet mouth). The touch screen will display a step up in the control graph as a result of the increase in frequency. Wait for the system to stabilise and make a note of the frequency value registered.

- Repeat the above operation 3 more times, until 2 ml of liquid have been introduced into the tank, each time making a note of the oscillation frequency value.

- Once there is 2 ml of liquid in the condenser, press the button "MAX" on the touch screen: in that moment, in the field "MAX.LEVEL" is saved the maximum value of the lavel sensor calibration. This value is not the sensor response with 2 ml of liquid, but the estimated response of the sensor for 10 ml of liquid in the condenser:

  $$\text{MAX.LEVEL (Hz)} = \text{Zero} + [(\text{FREC. 2 ml}) - \text{Zero}] \times 5$$

  If we set the sensor range in 2 ml, the increase in level inside the separator due the fall of a drop of liquid would represent a very high percentage regarding the maximum calibration value (2 ml), which would imply a very poor control procedure, with very sudden changes in the position of the control valve. To avoid this, the sensor range is extrapolated to 10 ml (once is tested the linearity between the sensor response and the level of liquid inside the condenser).

In this point, it is very important to check that the sensor range is configured at 10 ml: in the TOHO controller, _SLH parameter (in SET 2).

With these parameters, the level controller gives a REAL reading of the level of liquid inside the liquid / gas separator, in ml.

- By graphically plotting the sensor frequency against the amount of water present in the system, it can be verified that the system's response is perfectly linear, which enables a relationship to be established between oscillation frequency and the volume of water present in the tank.

- *Calibrating the sensor for any other fluid (X) (different from water):*

In order to verify linearity in the sensor's response to the quantity of liquid present in the system, Figure 4-32 and Figure 4-33 show the sensor's response to consecutive additions of 0.5 ml of different substances.

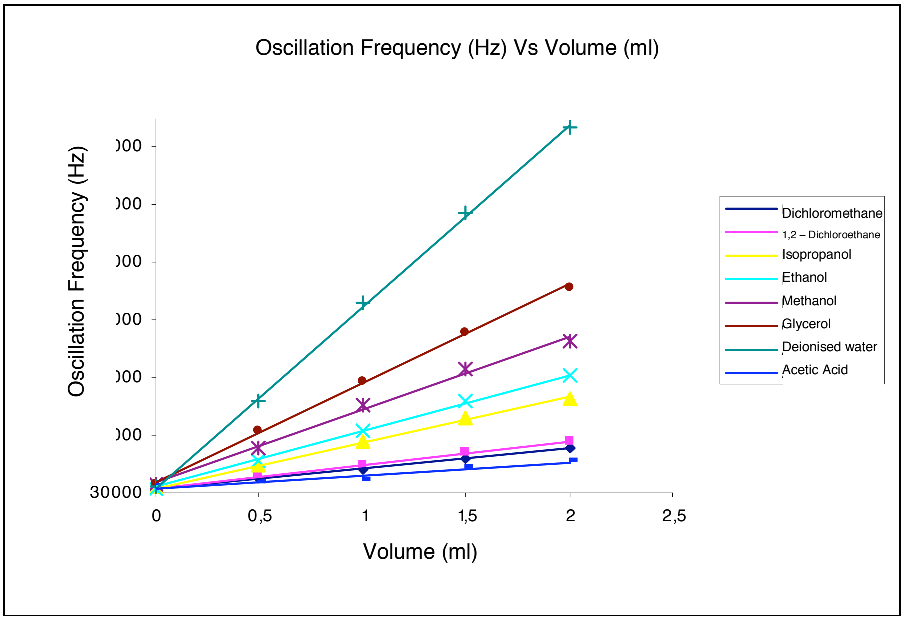


Figure 4-32

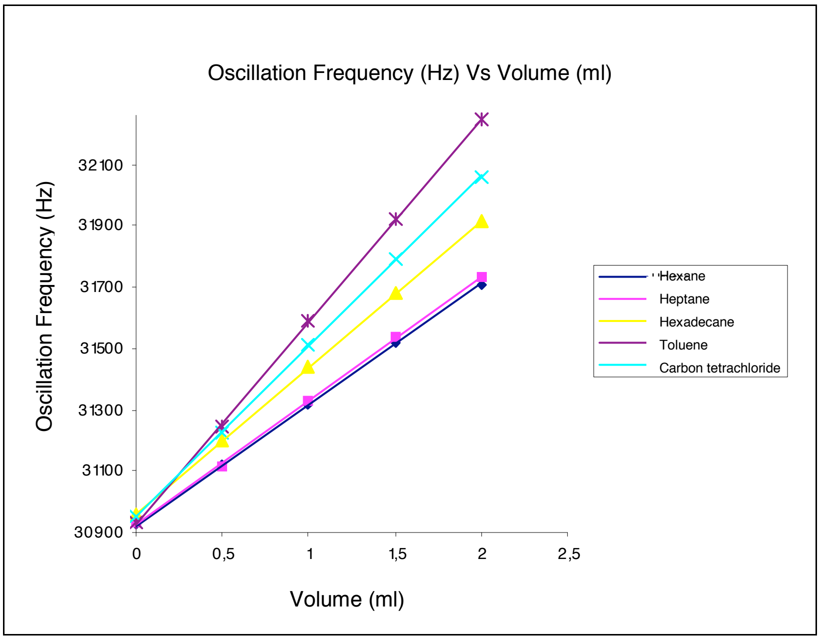


Figure 4-33

An analysis of the ratio existing between the oscillation frequency of the level sensor (difference between the response to the addition of 2 ml of a compound and the threshold) and its relative dielectric constant, shows that said response is almost perfectly linear for the whole range of dielectric constants (see Figure 4-34):

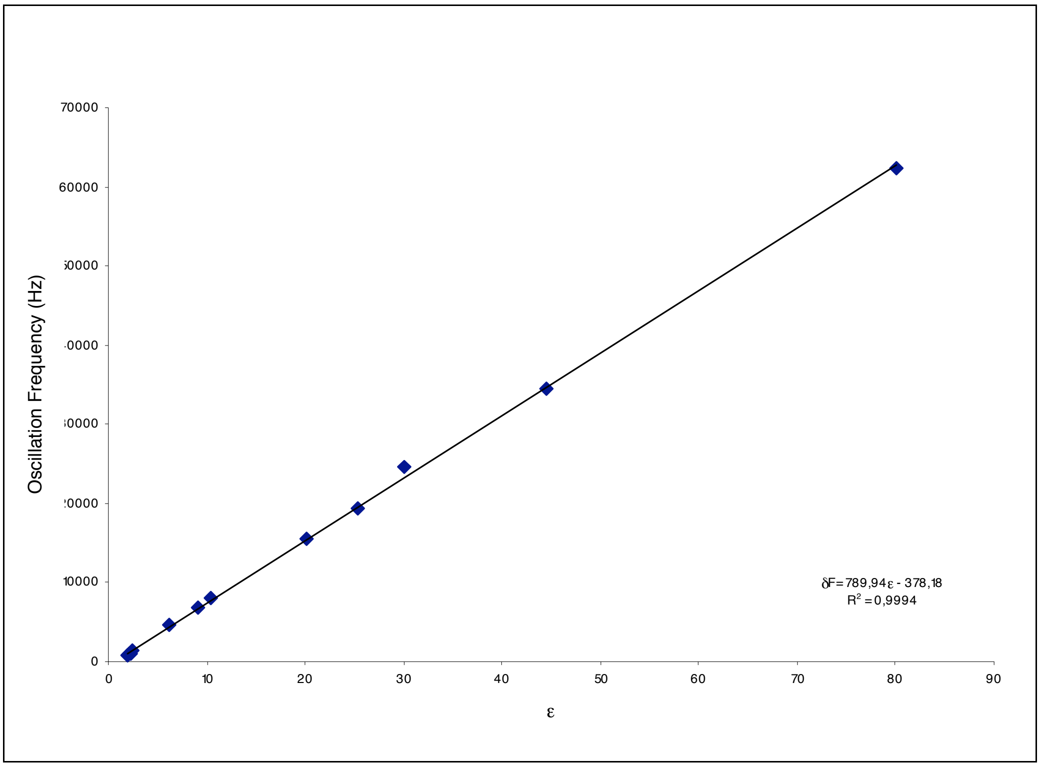


Figure 4-34

This performance by the sensor enables a calculation to be made of the expected response for a specific compound in terms of its dielectric constant, without the need to calibrate the system with the new compound and based on the calibration with water that is factory set. Accordingly, proceed as follows:

- Once the system is calibrated, select in the field “LIQUID K” on the “LEVEL SENSOR SETUP” screen the fluid that is going to condense on se liquid / gas separator. If that liquid is not in the list, select “OTHER” and set the dielectric constant (or an estimation) on the yellow field.
- Press the “**RETURN”** key to return to the level sensor calibration screen.
- In this moment, the field “MAX.LEVEL” changes, depending on the relation between the dielectric constants of the reference liquid (calibration liquid) and the actual.

At this point, the level controller gives a REAL reading of the level of liquid K inside the liquid / gas separator, in ml.

Table 4-5 lists the dielectric constants at 20ºC for different compounds:

COMPOUND	*FORMULA*	*ε (20ºC)*
Hexane	C_6H_{14}	1.89
Heptane	C_7H	1.92
Hexadecane	C_6H_{34}	2.05
Carbon tetrachloride	CCl_4	2.24
Toluene	C_7H_8	2.379
Acetic acid	$C_2H_4O_2$	6.17
Dichloromethane	CH_2Cl_2	9.08
1.2-Dichloroethane	$C_2H_4Cl_2$	10.42
Isopropanol	C_3H_8O	20.18
Ethanol	C_2H_6O	25.3
Methanol	CH_4O	30
Glycerol	$C_3H_8O_3$	44.52
Deionised water	H_2O	80.1

Table 4-5

The signal provided by the RC oscillator circuit (prior to being converted into an 4-20 mA analogue signal) may vary between:

- 31,000 – 90,000 Hz for water.
- 31,000 – 50,000 Hz for an alcohol.
- 31,000 – 32,000 Hz for a hydrocarbon.

In a situation such as this, a zero error on the instrument of, for example, 50 Hz, is insignificant in the case of fluids with a high dielectric constant, yet crucial in the case of fluids with a low dielectric constant. It is therefore important to be reasonably accurate when setting zero on the instrument and to do so the system must be empty, although it will have previously been moistened with the product to be measured. Once zero has been set on the instrument, it can be verified whether the instrument's output signal is directly proportional to the height of the liquid in the tank.

In the case of fluids with a low dielectric constant, it is important to be reasonably accurate when setting the instrument's zero or baseline. To do so the system must be empty, although it will have previously been moistened with the product to be measured.

For control purposes, the microprocessor converts the output signal into an analogue signal that can be understood by the control instruments. Typically, this is a 4-20 mA signal. Accordingly, all that is required is to indicate to the instrument that 31,000 Hz corresponds to an output of 4 mA and, for example, 50,000 Hz corresponds to an output of 20 mA. As of that moment, the instrument provides a 4-20 mA output signal proportional to the height of the liquid in the tank, without considering geometric issues in the design of the tank.

4.7. OTHER COMPONENTS ON THE MICROACTIVITY-REFERENCE UNIT

Connecting pieces, valves, instrumentation and other system components have been carefully selected. There follows a description of the more important ones, or those items that have undergone some form of modification in order to improve the system's performance.

4.7.1 PIPING

The selection of piping has taken several factors into account, such as temperature, pressure and type of compounds that are going to flow through it. The most widely used material is 316L stainless steel, with the maximum operating temperature for the pipe being that determined on the basis of the values for external diameter and thickness, using the ASME table for 316L stainless steel pipes for different temperatures.

For 316 type stainless steel without welding and at 316ºC, the maximum stress value permitted is 17000 psi. The geometry factor for a 1/8" pipe, with a wall thickness of 0.02", is 0.367. By applying the expression:

Maximum operating pressure = Maximum stress permitted x Geometry factor

A maximum operating pressure is obtained of 6239 psi (430 bar) for 1/8" pipes made of 316 stainless steel, with a thickness of 0.02" and at a temperature of 316 ºC.

The pipes selected that constitute the reactor are:

** Pipe:* TSS285.

- 1/8" pipe with internal diameter of 2.1 mm (0.085").
- Declared standards: EN–10204/3.1B – DIN50049/2.2.

** Pipe*: TSS120.

- 1/16" pipe with internal diameter of 0.50 mm (0.020").
- Declared standards: EN-10204/3.1B – DIN50049/2.2.

4.7.2 JOINTS AND VALVES

** Check valve for liquid feeding*: SG-SS-2C4-KZ-25.

- Located on the liquid inlet line to the reactor.
- 316 stainless steel valve with Kalrez sealing material.
- Maximum operating pressure and backpressure at 21ºC of 206 bar (3000 psig).
- Trigger pressure of 25 psi.
- Operating temperature of –23ºC at 191ºC.
- C_V = 0.10.

For a valve with a rated pressure trigger spring of 25 psig, true trigger pressure ranges between 21 and 29 psig. The minimum closing pressure is 17 psig.

** Long hexagonal male coupling*: SG-SS-HLN-2.00.

- 316 Stainless steel.
- Size both NPT male threads: 1/8". Length 2", interior diameter 0.19".

- Operating pressure as per ANSI for 31B.3 pressurised piping of 9400 psig.

This piece is used for connecting the joint on the liquid check valve SS-2C4-KZ-25 and the hot box, and its purpose is to transmit the temperature of the hot box to the non-return valve, as a result of its relatively high mass (solid piece). A series of Teflon pieces have been designed to reduce the flow section to the equivalent of a 1/16" pipe, as well as the dead volumes in the NPT type joints (see Figure 4-35).

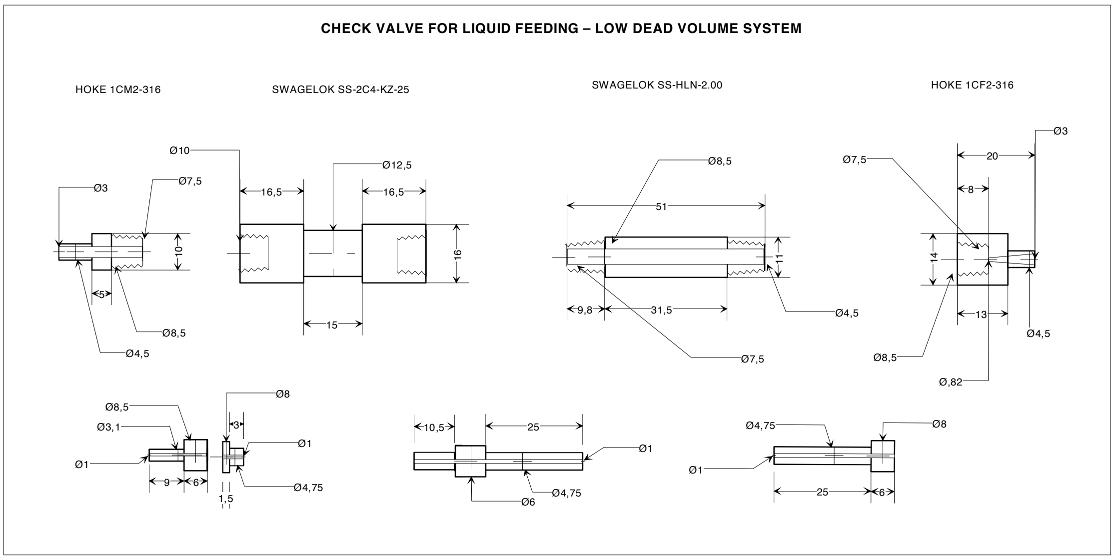


Figure 4-35

Figure 4-36 shows the Teflon pieces designed to eliminate the liquid non-return valve's dead volume.

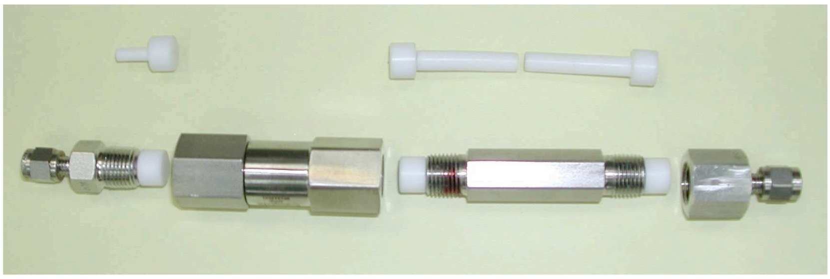


Figure 4-36

<u>** 3-port ("T") connection:*</u> VV-ZT2.

- 316 stainless steel. 1/8" pipe thread connection and orifice of 0.75 mm.
- Operating pressure of 400 bar.

In order to avoid standard wear and tear on the connecting pieces on a reactor (phenomenon that mainly occurs with T-shaped connections given the difficulty in using a "counter-spanner"), selection has been made of VICI-VALCO connecting pieces mounted onto the structure of the hot box, thereby enabling them to be manipulated with a single spanner.

<u>**Reduction element*</u> : VV-IZR21L.

- 1/8” VICI pipe thread reduction connection to 1/16” pipe thread.
- 316 type stainless steel.

** Bushing*: GY-2BU-316-PID.

- 316 type stainless steel. 1/8” extra-long threaded connection pipe.
- As per Standards ASTM A-182, ASTM A-479 and CMTR certificate.
- Operating temperatures ranging between –200ºC and 426ºC.

Hoke has custom-made extra-long bushing pieces for the Microactivity-Reference unit. They permit the inlet and outlet of process lines passing through the insulation separating the hot box and the thermal separation chamber from the rest of the mechanical assembly. These pieces are not commercially available.

** Non-return valve*: GY- 6133M2Y - Kalrez.

- 316 stainless steel. Joint in chemically compatible Kalrez (elastomer Teflon).
- As per Standards ASTM A-182, ASTM A-479 and CMTR certificate.
- Maximum operating pressure of 6000 psig at 70ºC (423 Kg/cm^2 at 21ºC).
- C_V = 0.3.

In order to avoid the multiple connections required for the feed arrangement of various gases and so as to favour the mixture of the same, a distributor has been designed in 316 stainless steel that reduces the number of joints and whose interior is fitted with a helicoidal feature that forces the gas stream through the strands on this propeller, favouring their mixing (see Figures 4-37 and 4-38).

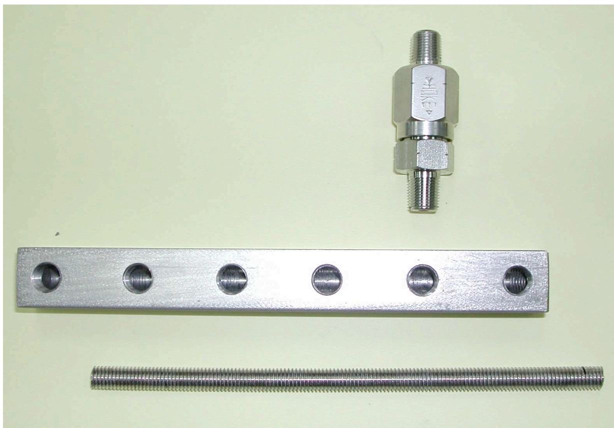


Figure 4-37

GAS MIXER

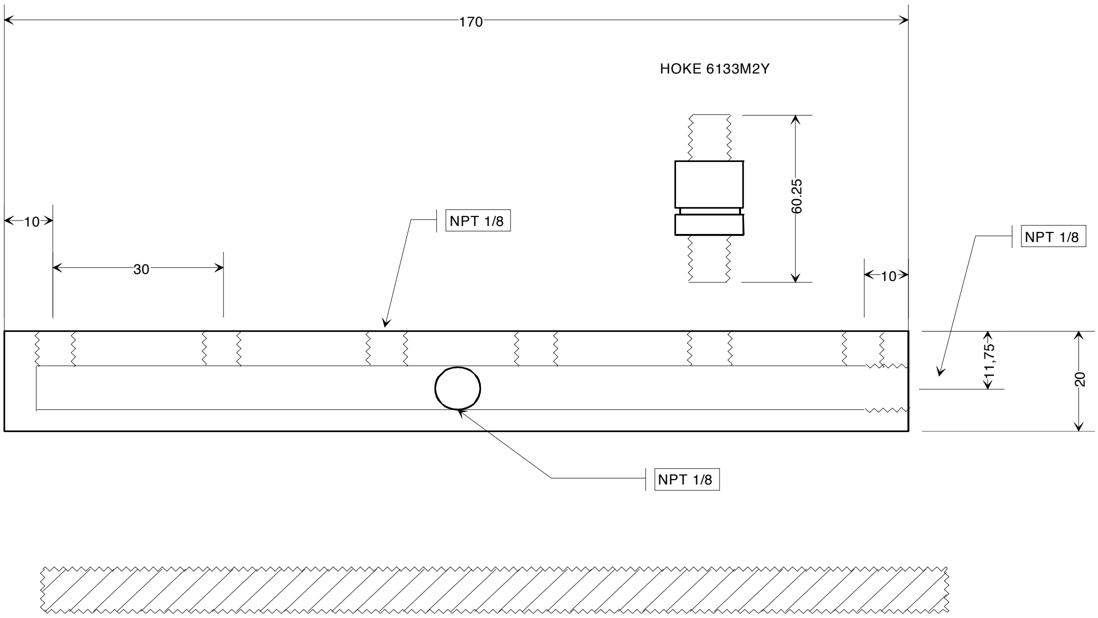


Figure 4-38

*_Reactor filters_*: VV-ZBUFR2F.

- Located on the reactor's gas inlet and outlet lines.
- 316 type stainless steel. 1/8" pipe connections. 2 mm (0.080") interior cavity.
- Porous panel of 10 µm in Hastelloy C (reference 10FR4HC).

4.7.3 INSTRUMENTATION

*_Pressure transducer_*: SWT A08.

Measurement range	0 -100 bar
Overpressure admissible	200 bar
Linearity	< 0.5% FS
Hysteresis and repeatability	< 0.1% FS
Thermal effect on zero	0.4% FS/10K
Thermal effect on the span	0.2% FS/10K
Operating temperature	(- 40) - 110ºC
Excitation potential	12 - 30 VDC
Output signal	4 – 20 mA
Power connection	M-12 connector, short-circuit protection
Pressure connection	G BSP 1/4" 316 stainless steel
Class of protection	IP65 (connector), IP67 (cable)
EMC	EN 50 081-1 and EN 50 082-2

Table 4-6

<u> Mass flow meters and controllers:</u>*. F201C – FAC– 11- X

A study has been performed on the features of different mass flow controllers, in terms of their reliability, accuracy, control valve design, operation in the lower run of the operating range and performance at high pressure, with the instruments made by the firm Hi-Tec Bronkhorst being selected as those best suited to a microactivity reactor.

Each controller is calibrated to perform the measurement of a specific compound, although the option exists to use them with compounds of a different factor to the unit's original calibration. When using another gas, special attention is to be paid to the elastomer's compatibility with the new process gas. Calibration at source is undertaken by means of equipment with NMI certification. The specifications of these instruments are as follows:

- AISI 316L stainless steel, 1/8" connections.
- 1% F.S. accuracy. 0.1% F.S. repeatability
- Operating range between 5% and 100%.
- Operating temperature between –10ºC and 70ºC.
- Input and output signal: 0-5 vdc.

<u> Temperature signal</u>*: TC-KIA-ID-0600-H0.

- K-type thermocouple, Inconel, 600 mm in length, mini-male high-temperature (220ºC) plug, 1.5 mm diameter, with temperature range between –200ºC and 1250ºC.
- Presents derivation and hysteresis as of 900ºC.
- Class 1 tolerance as per standards IEC 584.2: ±1.5ºC or ± (0.4%xT)ºC.

<u> Bypass valve</u>*: VV- AT36UWTY.

- 6 Ports with 2 positions, 1/8".
- Maximum operating pressure: 1500 psig.
- Maximum operating temperature 230ºC.
- Standard port diameter 0.75 mm.
- 316 stainless steel. Valcon T rotor material.
- 3" actuator–valve separation, with AT60 high temperature actuator.

4.8. CONTROL OF PROCESSES AND AUTOMATION

4.8.1 CONTROL LOOPS FOR THE MICROACTIVITY-REFERENCE

The Microactivity-Reference reactor uses closed loop controllers with proportional, integral and derivative type re-feed of the signal for the following control loops:

⇨ *Reaction temperature control:* The signal from the thermocouple located in the catalyst bed is assessed by the controller, whereby its 4-20 mA output signal is sent to a solid state "zero passage" type relay that regulates the power supplied to the oven in a proportional manner.

⇨ *Temperature control of the hot box*: The signal from the thermocouple located in the hot box is assessed by the controller, whereby its 4-20 mA output signal is sent to a solid state "zero passage" type relay that regulates the power supplied to the box's heater in a proportional manner.

⇨ There is a third zero passage relay for the possible temperature control of a heating pad for heating the gas outlet line, an evaporator, etc.

⇨ *Pressure control:* The signal from the pressure transmitter located upstream of the reactor is assessed by the controller, whereby its 4-20 mA output signal determines the position of the pressure control valve.

⇨ *Level control:* The signal from the capacitive signal installed in the liquid – gas separator is assessed by the controller, whereby its 4-20 mA output signal determines the position of the level control valve positioned at the base of the separator.

⇨ *Flow control:* The instruments used for dosing the gases into the reactor are in themselves flow controllers. Accordingly, the reactor's control system simply relays the 0-5 VDC control signals to the MFC. The signal recognises the 0-5 VDC output signal from the MFC and compares it to the order, triggering an alarm signal if they do not coincide.

Use has been made of controllers made by the firm TOHO, model TTM-005, for the control of temperature, pressure and level (4-20 mA input and output). These controllers cater for RS-485 digital communications for communicating with the computerised control system.

4.8.2 AUTOMATION

Before proceeding to the design of the printed circuit that contains the microprocessor that manages the system for the control and monitoring of the reactor, and which is what will manage the Microactivity-Reference reaction unit, a simulation was made of each one of the systems operating in the microactivity reactor.

4.8.3 SYSTEM SAFETY MANAGEMENT

⇨ **Safety status in the event of a power failure.**

As an initial safety measure, the controllers use a non-volatile memory to store the latest parameters introduced manually from the keyboard. After a power failure, the plant returns to these “safety” parameters or values, irrespective of what the latest values were that were sent to the computer.

As a precaution, the system’s “safety” values have to be re-entered after any manual modification of the set-point in operations performed by the operator, independently of the computerised control system.

⇨ **Temperature alarm.**

The controller’s upper limit absolute temperature alarm shuts down the control signal on the reactor oven and on the hot box heater and halts the operation of the MFC that the user has selected on the alarm set-up panel on the system’s touch screen. It also shuts down the operation of the HPLC pump, setting off an audible alarm as a warning signal for the operator, and triggers the safety system’s INH (inhibition) function, impeding the software changing session automatically.

⇨ **Operation of the hot box**

The hot box is fitted with a forced convection heater that consists of a turbine and heater. Its proper operation requires the turbine to be running when the heater is on, dissipating the heat and avoiding damage. This involves two systems working in parallel: provided that the controller sends a control signal higher than 5 mA to the “zero passage” solid state relay that regulates the heater’s power, the turbine starts operating, and even when this signal is not given, whenever the temperature in the hot box exceeds 40ºC the turbine will be running (The hot box controller’s upper limit absolute temperature alarm _E1H is set at 40 ºC). Accordingly, as the temperature drops, the turbine cools the hot heating cartridges.

⇨ **Door closed detector on hot box**

So as to disconnect the current supplied to the hot box heater when its door is open, there is an inductive sensor that detects whether it is open or closed. This function does not interrupt the operation of the turbine or act upon any other part of the system.

⇨ **Oven closed detector**

So as to disconnect the current supplied to the hot box heater and the reactor oven when the latter is open, there is a magnetic detector on the oven’s closing device that detects the position of the reactor’s moving section. The detection of an open oven interrupts the operation of the hot box heater, the supply of power to the oven and triggers the safety system’s INH (inhibition) function, impeding the software changing session.

If it becomes necessary to open the hot box door during a reaction procedure, this is to be done manually, without using the door opening button. Manual opening of the door will only interrupt the power supply to the box heater.

⇨ **Flow alarm**

This alarm is by percentage deviation over the set-point. If, for more than 10 s (or time set in the *Time Delay Alarm*) the set flow deviates by more than 10% from its value (percentage set on

the MFC set-up panel on the touch screen), the system interrupts the operation of the reactor oven, sets off an audible alarm (buzzer) to alert the operator and triggers the safety system's INH (inhibition) function, impeding changes in the operating session.

⇨ Pressure alarm

Absolute alarm, configured by parameter _E1H on the pressure controller. It acts upon the liquids pump and the MFC, halting the operation of those the user has selected on the pressure alarm set-up panel on the system's touch screen. It is self-locking, in order to avoid a repetitive cycle of the system in the event that its activity ceases even when the problem that triggered it has not been resolved. For example, in the event of a blockage in the porous plate, pressure will increase and the alarm will act upon the feed to the system, halting it. But this will bring the pressure below the alarm value, whereby the system will again start operating in a situation that will again produce the circumstances that will once again trigger the alarm. This is why once the pressure alarm has been triggered, the system will remain in stand-by until the operator proceeds manually to release this locking, an action that is to be performed after checking over the system and correcting the anomalous situation.

The safety system locks this situation until the system operator presses RESET on the pressure alarm, once the source of the problem has been located. The MFC will not operate until this RESET is pressed, even though the pressure value in the system has dropped below the value _E1H.

⇨ Level alarm

Alarm configured by the parameter _E1H on the level controller. It is absolute type and interrupts the operation of the liquids pump, triggers the audible alarm and the INH (inhibition) function as the upper limit set has been exceeded. This alarm is only available on equipment with liquid feed systems and a liquid – gas separator with level control.

⇨ Control valve failure

The final elements of pressure and level control are servo-positioned micro-regulating valves. A failure in the control system of these valves triggers an alarm of the same type as that triggered by their master control loops. A failure in the position of the pressure control valve generates a procedure similar to that generated by a pressure alarm, and a failure in the level control valve generates a procedure similar to that caused by an alarm in the separator's level controller.

⇨ INH Inhibition function

Whenever the INH function is activated by any one of the system's alarms, the Process@ control application automatically interrupts the performance of interconnected sessions. The system's control program will remain in stand-by until the operator manually implements the change of session once the cause of the alarm has been resolved.

⇨ External alarm

A signal forthcoming from an outside system for detecting gases, fire or such like may trigger a general alarm in the system that involves disconnecting the hot box heater, the reaction oven, all the MFC, the liquid pump, triggering the audible alarm (buzzer) and generating the system's inhibition signal.

SUMMARY TABLE OF THE ALARM FUNCTIONS

ALARM	TYPE	REACTOR OVEN	HOT BOX HEATER	HOT BOX CONVECTOR	REACTANTS MFC´s	INERTS MFC´s	LIQUIDS PUMP	INH	BUZZER
REACTION TEMPERATURE	ABS	OFF	OFF		(1)			ON	ON
HOT BOX TEMPERATURE	ABS			ON					
PRESSURE CONTROL	ABS				(1)			ON (2)	ON
LOSS OF LOAD	ABS				(1)			ON (2)	ON
LEVEL CONTROL	ABS						OFF	ON	ON
MASS FLOW CONTROLLERS	DEV	OFF						ON	ON
DOOR-OPEN DETECTOR	Relay		OFF						
OVEN-OPEN DETECTOR	Relay	OFF	OFF					ON	
EXTERNAL ALARM	Relay	OFF	OFF		(3)			ON	ON
PRESSURE SERVO-CONTROL	Relay				OFF	OFF	OFF	ON	ON
LEVEL SERVO-CONTROL	Relay						OFF	ON	ON

Table 4-7

(1) Actions defined by the user in the set-up menu for the reaction pressure and temperature alarms on the touch screen of the Microactivity-Reference.

(2) The inhibition session is activated in the event of a pressure alarm, with this status persisting until the user resets the alarm on the alarm panel on the touch screen. (In all other alarm scenarios, the inhibition function is deactivated automatically once the system's parameters return within the established control limits).

(3) The actions of the external alarm on the MFC and the liquids pump will be the same as those configured by the user on the touch screen for a pressure alarm.

5. PROCESS@ CONTROL SOFTWARE

5.1. INTRODUCTION

Process@ control software is an application for data supervision and acquisition designed for systems based on digital communications between process hardware and a personal computer. Use of this application permits data acquisition and the remote control of one or several Microactivity-Reference units via Ethernet type communications.

5.2. INSTALLATION OF PROCESS@

The installation of Process@ control software involves the following steps:

- Insert the CD-ROM containing the Process@ control into the CD reader drive.
- It is advisable to close all other Windows applications that are running.
- Run the “setup.exe” file on the CD. To do so, use Windows Explorer or the “Run” option on the Start Menu.
- The installation assistant will take you through the various stages of the installation.
- Finally, you will be required to reboot the system.
- The installation assistant will create a shortcut to Process@ on the Desktop.

5.3. COMMUNICATION PARAMETERS CONFIGURATION

The Microactivity-Reference unit is configured, by default, with the communication parameters to connect it directly to the control PC. The unit user has to check that these parameters are the appropriated ones in both units:

5.3.1 COMMUNICATION PARAMETERS OF THE MA-REF UNIT

These parameters are shown in the screen “Communication Setup” of the touch screen main menu (see section 4.5.2 of this manual). By default, the unit is configured with these parameters:

IP ADDRES:	**192.168.0.5**
GATEWAY:	**192.168.0.1**
IP MASK:	**255.255.255.0**
TCP PORT:	**1234**

When the connection is made via Ethernet, the user has to modify these parameters to adapt them to the local net.

5.3.2 COMMUNICATION PARAMETERS OF THE PERSONAL COMPUTER

To check these parameters in Windows operative system, proceed as follows:

- Click on the icon “**Network Connections**” with the right button of the mouse and select “**Properties**”.

- Click on the icon "**Local Area Connection**" with the right button of the mouse and select "**Properties**" (Figure 5-1).

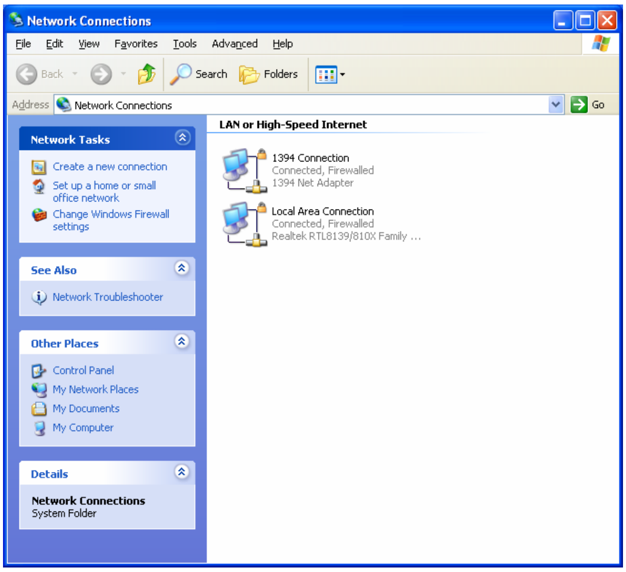


Figure 5-1

- Select "**Internet Protocol (TCP/IP)**" and click on "**Properties**".

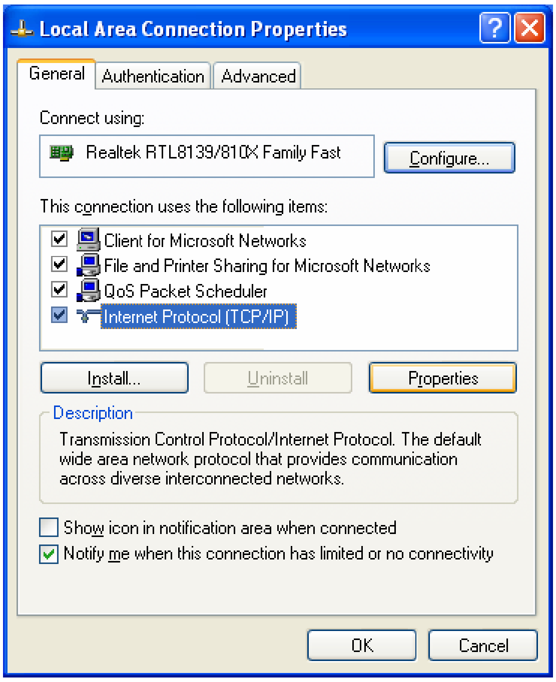


Figure 5-2

- Set the communication parameters:

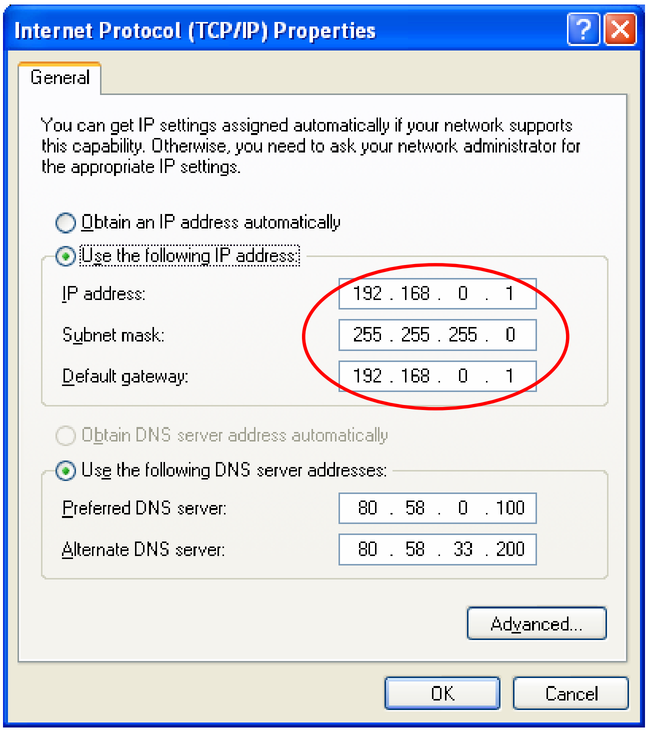


Figure 5-3

IP Address:	**192.168.0.1**
Subset mask:	**255.255.255.0**
Default Gateway:	**192.168.0.1**

5.4. USE OF PROCESS@

5.4.1 STARTING PROCESS@ APPLICATION

To begin using the application, click on the application (Figure 5-4):

Figure 5-4

The main functions bar will then be displayed. This is the starting point for use of the entire application.

5.4.2 THE FUNCTIONS BAR

The functions bar is the first screen to appear when beginning to use the Process@ application, and it is the tool that provides access to all the other functions for starting and ending acquisition, creating experiments, session control and so forth (Figure 5-5).

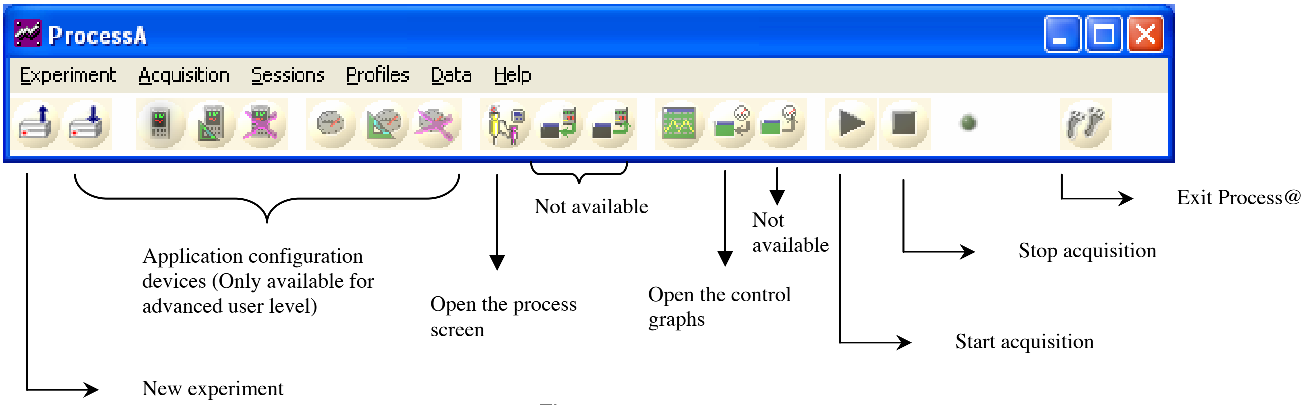


Figure 5-5

5.4.3 THE PROCESS SCREEN

Upon opening the Process@ application and creating an experiment (see section “Performing an experiment”) a screen will display a diagram of the Microactivity-Reference unit, featuring all the virtual devices that make up the control panel and which, logically, correspond to the real process hardware. Two-way digital communication takes place between them:

- From the process information to the data acquisition application.
- From the virtual or “desired” process information, held in the computer’s virtual devices, to the hardware as depicted on the control panel.

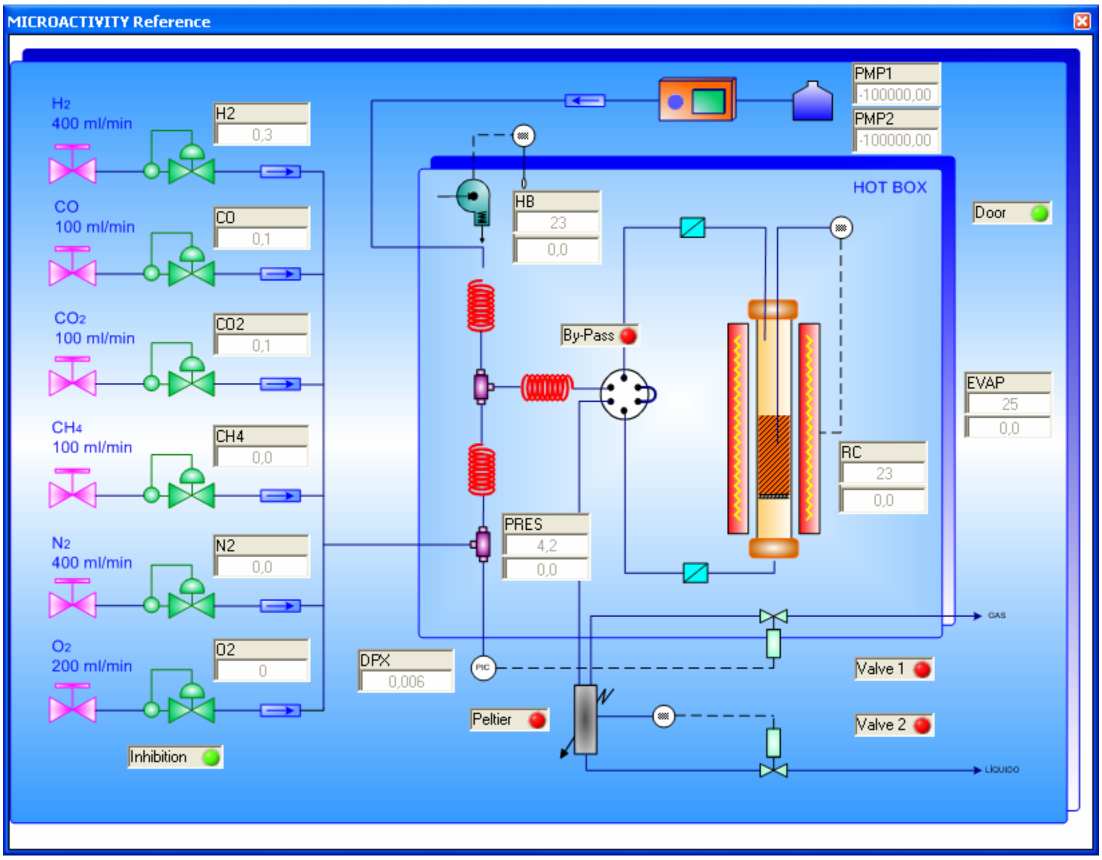
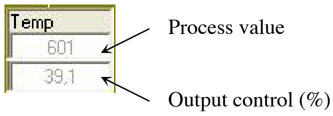


Figure 5-6

The plant's different devices are represented by means of the following kind of displays (the displays presented on the process screen will depend on the specific configuration of the Microactivity-Reference unit):

1. Displays that show the variable's current process value:
   - Mass flow controllers of the inlet gases
   - Liquid flow pump
   - Scale for collection of condensable products
   - Mass flow meter for outlet gases.
   - Pressure transducer (DPX)

2. Displays that show the variable's process value and set-point:
   - Reactor temperature
   - Hot box temperature
   - System pressure
   - Level in liquid – gas separator
   - Evaporator



3. Displays that show the on/off status of the device:
   - By-pass
   - Peltier
   - Door
   - Inhibition function
   - VICI extra valves (V1, V2, etc.)

The status of the devices represented by displays of this kind is as follows:

STATUS	DEVICE			
	BY-PASS	PELTIER	DOOR	INHIBITION
	By-pass: reactor isolated	Condenser cooled	Open	Activated: The programmed sequence of sessions is interrupted
	No by-pass	Condenser without cooling	Closed	Deactivated: The programmed sequence of sessions is followed

Table 5-1

The process screen displays the process status at any given moment and, therefore, it is not possible to modify the parameters of the devices on it (in the version Process@ 1.0). To do so, either carry out the modification on the Session Configuration Panel or directly modify the hardware (directly via the TOHO controllers or by means of the touch screen that is part of the Microactivity-Reference unit).

For opening a new process screen, click on the button .

5.4.4 THE PROCESS CONTROL GRAPHS

The process control graphs are graphic windows in which the current process values acquired by the control software are plotted in real time.

When a new experiment is created, two graphs will automatically appear in the upper right-hand corner of the PC screen. In these, the x-axis represents the relative time elapsed since the start of data acquisition and the y-axis represents:

- The process values of the controlled variables (upper graph).
- The control outlet of the controlled variables (lower graph).

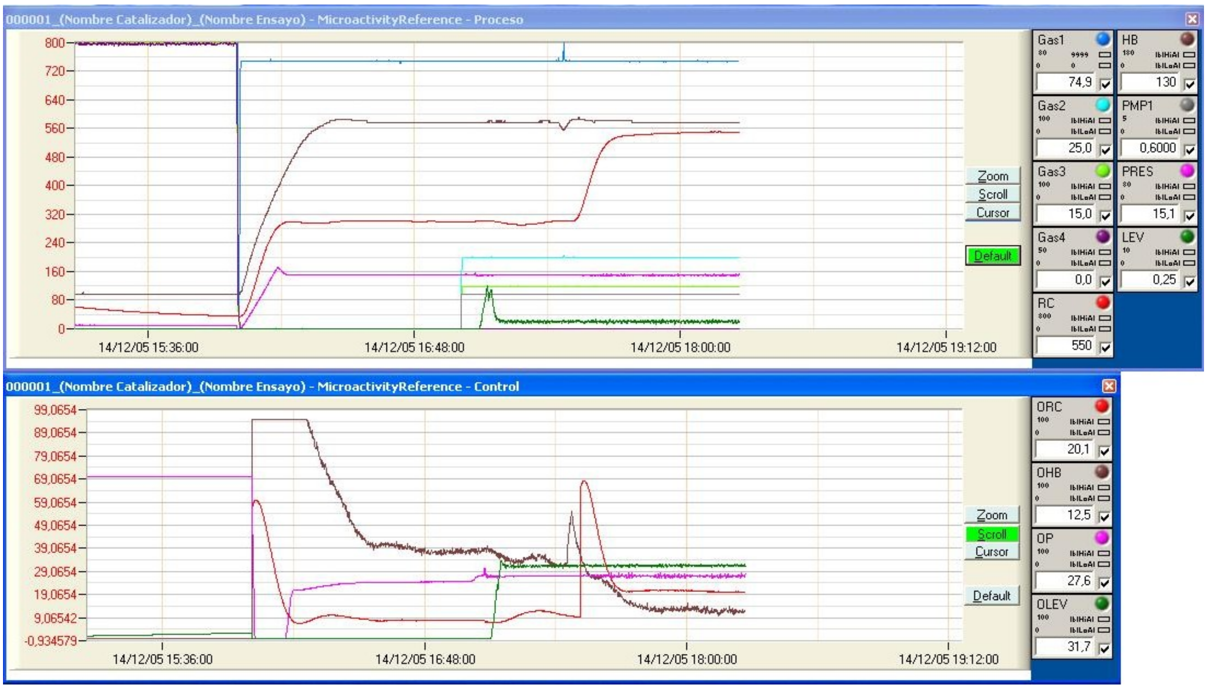


Figure 5-7

As soon as data acquisition starts, the graphs will begin to plot the process values of the controlled variables. Each one of these variables is represented by a display (to the right of the control graphs), with the following appearance:

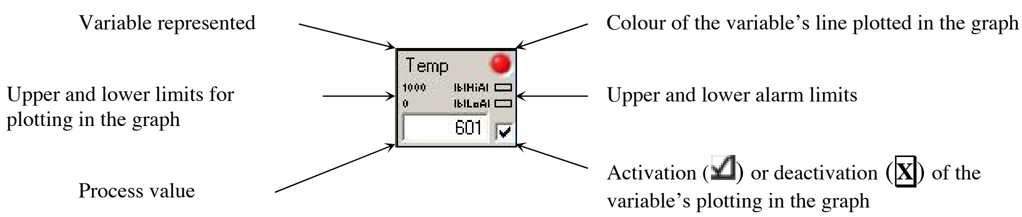


Figure 5-8

By moving the cursor over the display and clicking the right button on the mouse, access is gained to the set-up window for the chosen variable. The set-up options are as follows:

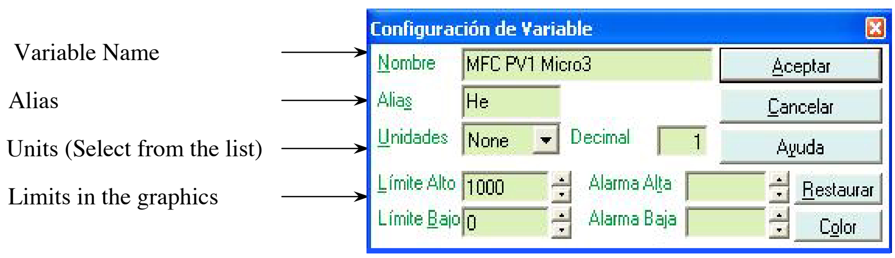


Figure 5-9

- **Decimal**: No. decimals for the variable
- **Upper Alarm**: Value of the variable above which the alarm will be triggered
- **Lower Alarm**: Value of the variable below which the alarm will be triggered
- **Colour**: Choose the colour of the variable's line in the graph.
- **Restore**: This restores the variable's default setting

All changes made to the setting of the variables' displays are stored in the memory by clicking on "**Accept**".

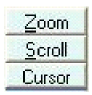
Each one of the variables in the process graphs is depicted by means of a continuous line, as per the colour of its corresponding display, which shows the evolution of said variable over time.

The X and Y axes on the graph may be modified as follows:

- Y-axis: this shows the upper and lower limits configured for each one of the variables. The axis's numerical scale will at all times feature the colour of the variable whose limits it is featuring. To switch from one variable to another, simply click the mouse's left button on the display of the variable whose limits are to be called up.

- X-axis: this shows a window of relative time elapsed since the start of data acquisition, with the format: **dd/mm/yy h:min:sec**. The size of this time window may be modified by clicking on the x-axis with the mouse's left button.

The three function keys that are to be found on the right of the process graphs allows for configuring their display setting:



ZOOM: It increases the size of the selected window: Keep the left button on the mouse depressed and draw the required box

SCROLL: It allows for dragging the graph backwards and forwards: Keep the left button on the mouse depressed and drag the graph

CURSOR: It shows the exact value of the variable at the point selected with the mouse (click the mouse's left button on the control graph)

DEFAULT: To come back to the default display

Each one of the functions is activated by clicking the mouse's left button on them, and they remain activated until a further click is made, whereupon the graphs' normal setting is restored.

For opening a new control graph, press the button .

5.5 PERFORMING AN EXPERIMENT

5.5.1 CREATING AN EXPERIMENT

To create an experiment, click on the icon on the process@ functions bar, or select "**New Experiment**":

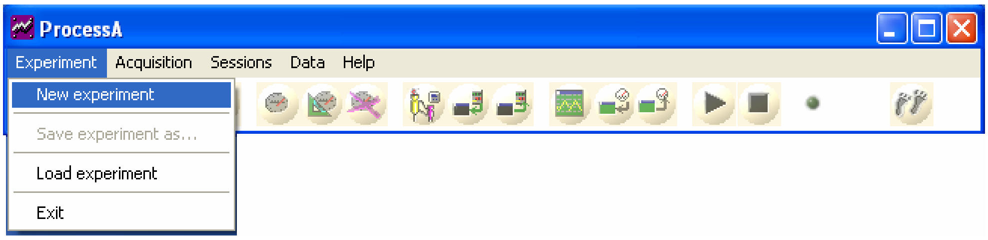


Figure 5-10

The experiment definition screen will be displayed:

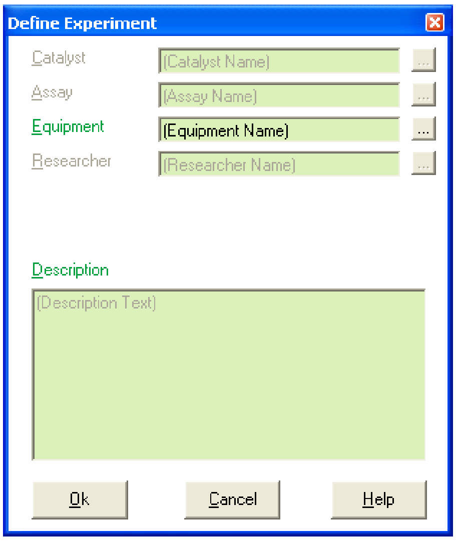
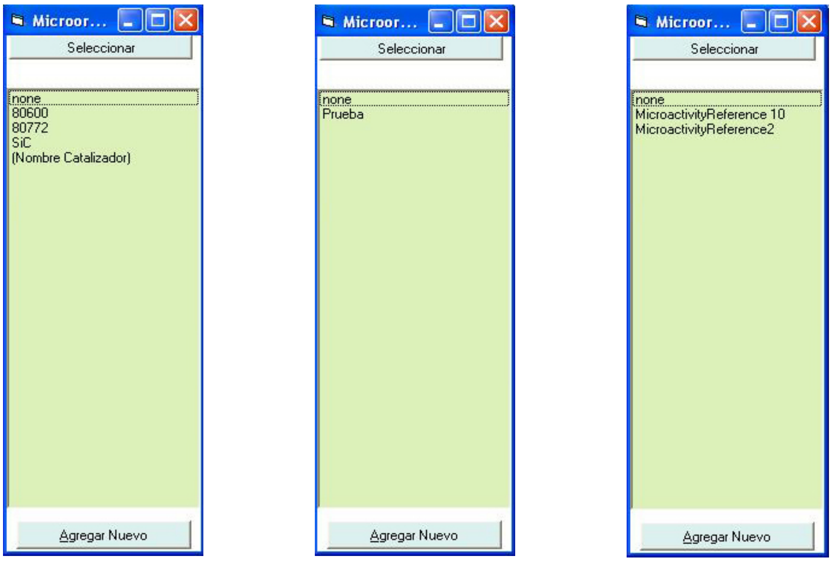


Figure 5-11

This screen allows for setting the following characteristics or parameters of the experiment that is to be carried out:

- Name of the catalyst used
- Name of the test
- Equipment in which it is to be performed
- Name of the head researcher

All these parameters may be set by clicking on the icon that is to the right of each one of them, thereby accessing the following screens:



Screen for defining the name of the catalyst

Screen for defining the name of the test

Screen for defining the equipment

Figure 5-12

Each one of these screens will call up a list with the different catalyst and test names, as well as the different equipment or devices that are controlled by the system (1, 2, etc.). To select each one of these, simply use the left button on the mouse to select the desired option and click on the button "**Select**". In the event that the desired name is not featured on the list, a new one may be created by means of the function "**Add new**". Once all the fields have been selected, click on "**Accept**" on the experiment definition screen. (This screen includes a space where, on an optional basis, a description of the experiment or some other further observation may be included).

The choice of catalyst, test and equipment will determine the name of the experiment that is to be performed, being expressed as follows (e.g.):

000029_SiC_Trial_MicroactivityReference10

Name of equipment

Test

Catalyst

No. of experiment performed in equipment (automatic)

5.5.2 DATA ACQUISITION

To start the system acquiring the plant's parameters, click on the button that is to be found on the functions bar: the system will start registering the parameters of the Microactivity-Reference, as well as plotting the control graphs. This process may be paused or stopped whenever the user wishes, using the respective keys and on the functions bar.

The frequency of acquisition of the system's parameters may be established by modifying the sampling period (time elapsed between two successive data acquisitions), which is accessed by

clicking on the tab “**Acquisition**” on the functions bar and selecting the function “**Sample Rate**”, as shown in Figure 5-13:

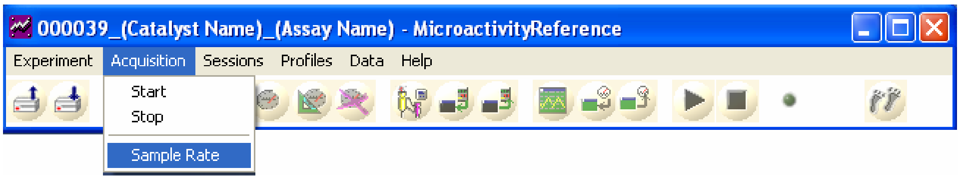


Figure 5-13

The following screen will be displayed (Figure 5-14), in which the desired sampling period can be set in milliseconds. It is advisable to use sampling periods of 5 - 10 seconds (5000 – 10000 ms). Changes made to this parameter will not be stored in the system until the “**Accept**” button is clicked.

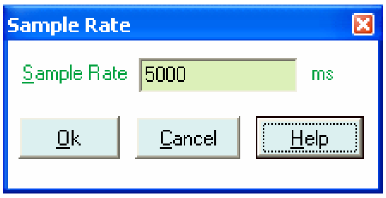


Figure 5-14

With the acquisition of parameters, the control PC will store the different parameters registered by the Microactivity-Reference unit in its memory, but no experiment will be sent. In order to send an experiment or programmed sequence of sessions to the unit, proceed from the Session Configuration Panel, as explained in the following section.

5.5.3 THE SESSION CONFIGURATION PANEL

The configuration of the process sessions is the next step to be performed by the user once the name of the experiment has been defined (see previous section) and data acquisition has started, with “sessions” understood to be each one of the process states or steps, in each one of which each variable has a specific set-point.

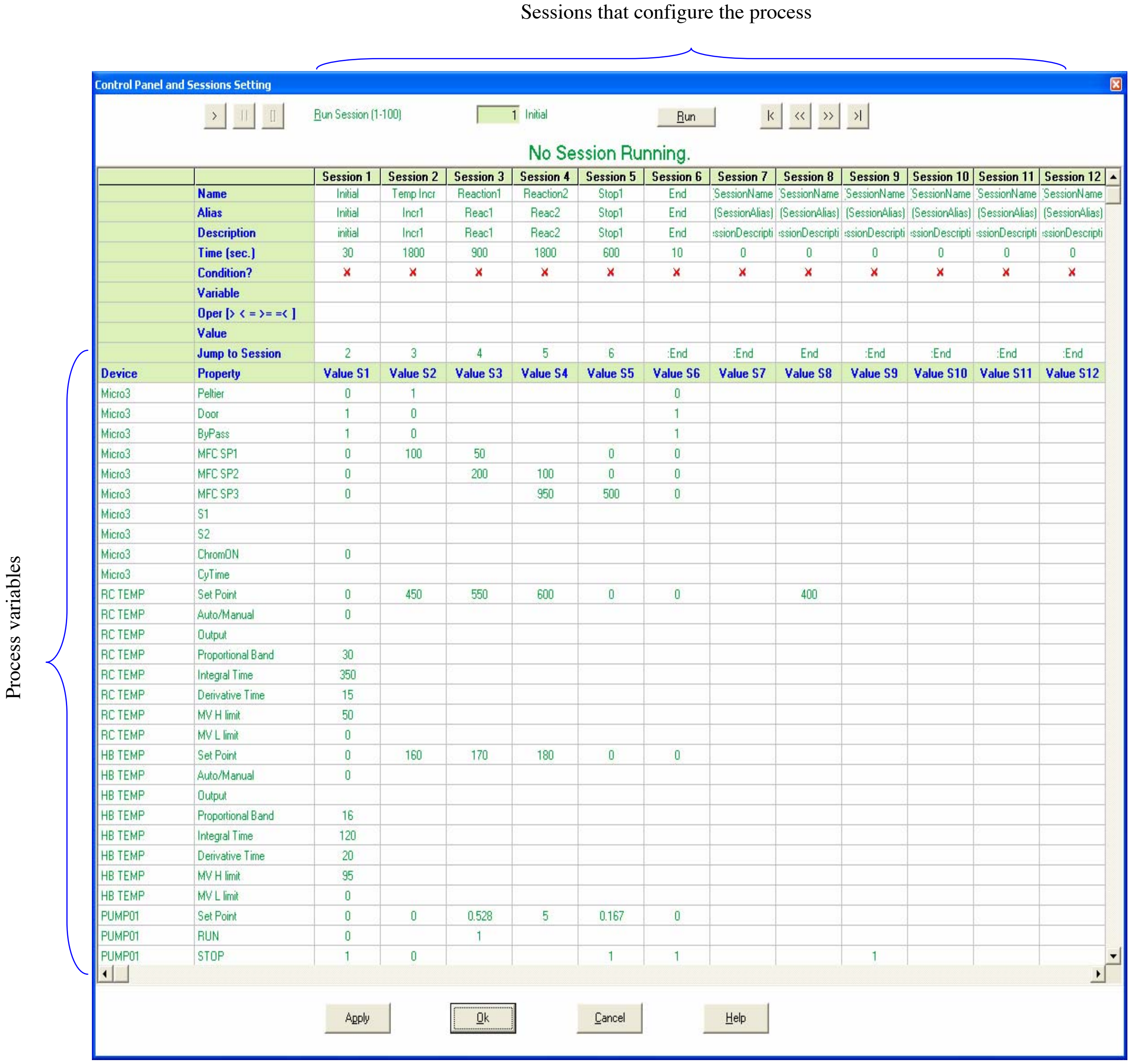
The Process@ application allows for the creation and configuration of as many sessions as required, which are to be carried out progressively, as defined by the user. The linking of these sessions or steps will make the process develop along the path desired and defined by the user.

The session configuration panel is accessed as follows:

1. On the functions bar, use the mouse’s left button to click on the tab “**Sessions**”.
2. In the new window displayed, select the option “**Configure Sessions**”.

Figure 5-15

Display will immediately be made of the Session Configuration Panel, which is presented below. (Figure 5-16).



		Session 1	Session 2	Session 3	Session 4	Session 5	Session 6	Session 7	Session 8	Session 9	Session 10	Session 11	Session 12
	Name	Initial	Temp Incr	Reaction1	Reaction2	Stop1	End	SessionName	SessionName	SessionName	SessionName	SessionName	SessionName
	Alias	Initial	Incr1	Reac1	Reac2	Stop1	End	(SessionAlias)	(SessionAlias)	(SessionAlias)	(SessionAlias)	(SessionAlias)	(SessionAlias)
	Description	initial	Incr1	Reac1	Reac2	Stop1	End	ssionDescripti	ssionDescripti	ssionDescripti	ssionDescripti	ssionDescripti	ssionDescripti
	Time (sec.)	30	1800	900	1800	600	10	0	0	0	0	0	0
	Condition?	✗	✗	✗	✗	✗	✗	✗	✗	✗	✗	✗	✗
	Variable												
	Oper [> < = >= =<]												
	Value												
	Jump to Session	2	3	4	5	6	:End	:End	End	:End	:End	:End	:End
Device	Property	Value S1	Value S2	Value S3	Value S4	Value S5	Value S6	Value S7	Value S8	Value S9	Value S10	Value S11	Value S12
Micro3	Peltier	0	1				0						
Micro3	Door	1	0				1						
Micro3	ByPass	1	0				1						
Micro3	MFC SP1	0	100	50		0	0						
Micro3	MFC SP2	0		200	100	0	0						
Micro3	MFC SP3	0			950	500	0						
Micro3	S1												
Micro3	S2												
Micro3	ChromON	0											
Micro3	CyTime												
RC TEMP	Set Point	0	450	550	600	0	0		400				
RC TEMP	Auto/Manual	0											
RC TEMP	Output												
RC TEMP	Proportional Band	30											
RC TEMP	Integral Time	350											
RC TEMP	Derivative Time	15											
RC TEMP	MV H limit	50											
RC TEMP	MV L limit	0											
HB TEMP	Set Point	0	160	170	180	0	0						
HB TEMP	Auto/Manual	0											
HB TEMP	Output												
HB TEMP	Proportional Band	16											
HB TEMP	Integral Time	120											
HB TEMP	Derivative Time	20											
HB TEMP	MV H limit	95											
HB TEMP	MV L limit	0											
PUMP01	Set Point	0	0	0.528	5	0.167	0						
PUMP01	RUN	0		1									
PUMP01	STOP	1	0			1	1			1			

Figure 5-16

The following parameters are to be set for each one of the sessions:

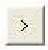
- **Name**: Name of the session
- **Alias**.
- **Description** (optional): Brief outline of the session.
- **Time (sec.)**: Time, expressed in seconds, the session is going to last. Once this time has elapsed, the system will go on to carry out the next session, which will be the one specified in the box "Index next session".
- **Evaluate condition?** (Option not available in the version Process@ 1.0). 2 options (switch from one to the other with the mouse's left button):

- ☒ : The system will not evaluate any condition: it will jump to the next session when the established session time has elapsed.
- ☑ : The system will evaluate the condition set forthwith. The jump to the next session (specified in “Index next session”) will occur in the event of any one of the following situations:
  - When the specified condition has been fulfilled (e.g.: $T_{reactor}$ > 500ºC).
  - When the established session time has expired.

- **Variable**: Variable to be evaluated in the condition. (e.g.: $T_{reactor}$).
- **Operator**: Operator that is to be used in the condition (>, <, =, >=, <=).
- **Value**: Value of the variable upon which the condition is set (e.g.: 500ºC)
- **Index of next session**: No. of the session with which the link-up is to be made (1, 2, 3 etc.)

The next step is to define the status that is to apply to each variable in the different process sessions. The variables, with their different options, are as shown in Table 5-1.

The upper part of the session configuration panels features the session control keys that are detailed below:

 PLAY: Launch session no. 1.

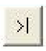
 PAUSE: Pause the sequence of sessions (session time continues to run, but no jump is made to the next session).

 STOP: Stop the sequence of sessions.

 First session (Display is made in the window of the experiment’s first session (no. 1), but this is not performed until the button “PLAY” is pressed).

 Prior session (Display is made in the window of the experiment’s prior session, but this is not performed until the button “PLAY” is pressed).

 Subsequent session (Display is made in the window of the experiment’s subsequent session, but this is not performed until the button “PLAY” is pressed).

 Final session (Display is made in the window of the experiment’s final session (no. 100), but this is not performed until the button “PLAY” is pressed).

DISPOSITIVE	PROPERTY	CHARACTERISTICS
Micro 3	Peltier	Condenser refrigeration: • 0: OFF • 1: ON
Micro 3	Door	Hot box door: • 0: Closed • 1: Opened
Micro 3	ByPass	By-pass valve position: • 0: Reactor position • 1: Reactor by-passed
Micro 3	MFC SP1	Gas 1 flow (ml/min)
Micro 3	MFC SP2	Gas 2 flow (ml/min)
Micro 3	MFC SP3	Gas 3 flow (ml/min)
Micro 3	MFC SP4	Gas 4 flow (ml/min)
Micro 3	MFC SP5	Gas 5 flow (ml/min)
Micro 3	MFC SP6	Gas 6 flow (ml/min)
Micro 3	S1	Actuator / Switch S1: • 0: Deactivated • 1: Activated
	S2	Actuator / Switch S1: • 0: Deactivated • 1: Activated
Micro 3	S3	Actuator / Switch S1: • 0: Deactivated • 1: Activated
Micro 3	ChromOn	Chromatography cycle: • 0: Deactivated • 1: Activated
Micro 3	Cytime	Time for chromatography cycle (time between two analysis), in seconds.
REACTOR	SV1 Ramping Time	Slope for set point (ºC/min)
REACTOR	Set Point	Reactor temperature set point (ºC)
REACTOR	Auto / Manual	Control mode: • 0: Automatic (Run) • 1: Manual (Man)
REACTOR	Output	Output control % (in manual mode)
REACTOR	Proportional Band	Proportional band
REACTOR	Integral Time	Integral time
REACTOR	Derivate Time	Derivate time
REACTOR	MV H Limit	Upper limit of the output control
REACTOR	MV L Limit	Lower limit of the output control
HOT BOX	Set Point	Hot box temperature set point (ºC)
HOT BOX	Auto / Manual	Control mode: • 0: Automatic (Run) • 1: Manual (Man)
HOT BOX	Output	Output control % (in manual mode)
HOT BOX	Proportional Band	Proportional band
HOT BOX	Integral Time	Integral time
HOT BOX	Derivate Time	Derivate time
HOT BOX	MV H Limit	Upper limit of the output control
HOT BOX	MV L Limit	Lower limit of the output control
PRESSURE	Set Point	Pressure set point (ºC)
PRESSURE	Auto / Manual	Control mode: • 0: Automatic (Run) • 1: Manual (Man)
PRESSURE	Output	Output control % (in manual mode)
PRESSURE	Proportional Band	Proportional band
PRESSURE	Integral Time	Integral time
PRESSURE	Derivate Time	Derivate time
PRESSURE	MV H Limit	Upper limit of the output control
PRESSURE	MV L Limit	Lower limit of the output control
LEVEL	Set Point	Level set point (ºC)
LEVEL	Auto / Manual	Control mode: • 0: Automatic (Run) • 1: Manual (Man)
LEVEL	Output	Output control % (in manual mode)
LEVEL	Proportional Band	Proportional band
LEVEL	Integral Time	Integral time
LEVEL	Derivate Time	Derivate time
LEVEL	MV H Limit	Upper limit of the output control
LEVEL	MV L Limit	Lower limit of the output control
PUMP	Set Point	Liquid flow (ml/min)
PUMP	Run	Turn on the pump (put “1”)
PUMP	Stop	Turn off the pump (put “1”)
SCALE	Rezero	Set zero in the scale

Table 5-1

When one session is linked up to another, the system will maintain the values set for all those variables that are not modified in the change of session, whereby it will not be necessary to re-introduce their value in the new session.

Sessions may be linked up with each other in a random manner, as per the user's wishes, without having to follow a numerical order (1, 2, 3, etc.). However, this system is not recommended as, in this case, monitoring of the reaction on the session configuration panel becomes much more complicated and increases the risk of incorrect programming of the sessions, as well as of becoming caught up in endless loops.

Once the session configuration panel has been completely filled in, press the key "**Apply**" that is to be found on the lower part to store the parameters entered in the system's memory. With this action, the panel will remain active on the screen, thereby allowing for better monitoring of the experiment and rapid access to the modification of session parameters. PLEASE NOTE! These modifications will not be registered in the application until the key "**Apply"** is pressed. Pressing the key "**Accept**" closes the session configuration panel.

5.5.4 DATA PROCESSING AND GRAPH PRESENTATIONS

The values of the variables acquired by Process@ may be exported to an Excel spreadsheet for subsequent processing. The procedure to be followed is as follows:

5.5.4.1 Recovering data from the experiment in process

To recover data from an experiment that has just been run in Process@ and which remains active (the control software has not been closed), carry out the following steps:

1. On the functions bar, use the mouse's left button to click on the tab "**Data**".

2. Select the option "**Variable**" (Figure 5-17).

Figure 5-17

3. A window like the one shown in Figure 5-18 will open, where the user may select the variables whose data are to be recovered (e.g.: RCTemp, Pressure, etc.). Clicking on the red crosses, the user selects the desired variables.

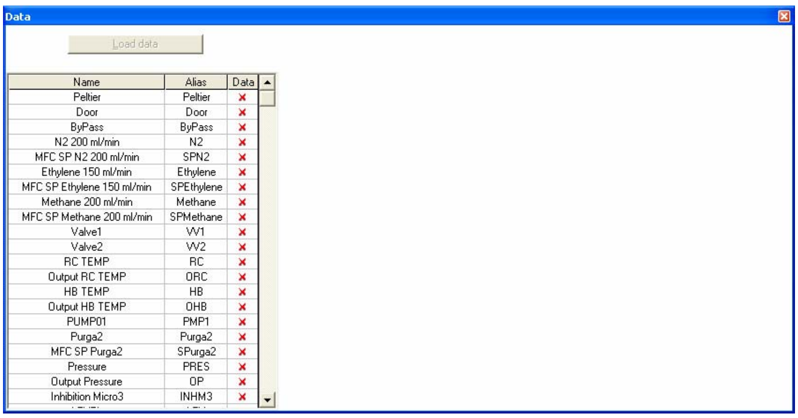


Figure 5-18

4. Once the variables have been selected, click on “**Load data**” and data columns will appear in the right-hand side of the window, displaying the following:
    a. On the left, the “time” variable, in the format **dd/mm/yy h:min:sec**.
    b. On the right, the values acquired of the selected variable.

5. Click on the right button of the mouse, select “**Copy all data to clipboard**”. Now these data can be pasted in any data sheet (Excel, Origin, Txt, etc). The format of the Excel columns must have the same format for each cell (number with/without decimals, date, time, etc.) as the data that have been imported.

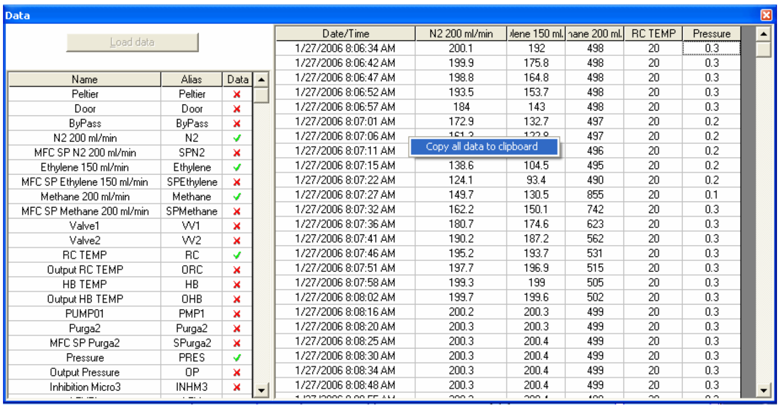


Figure 5-19

5.5.4.2 Recovering data from prior experiments

For recovering data from prior experiments, the user should proceed as follows:

- ***Open a new Process@ session: if there is an experiment running, the user can open a second Porcess@ session at the same time, clicking on the application icon (Figure 5-4).***

- In Process@ functions bar, select “**Experiment / Load Experiment**” (Figure 5-20)

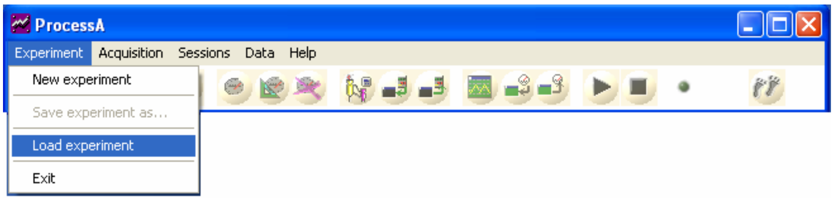


Figure 5-20

- Select the experiment to recover: *(Series No.)_(Catalyst Name)_(Test Name).adb*

- ***When the user is loading an old experiment in a Process@ session, it is no possible to acquire data or sending programmed sessions to the reactor with this session (but it is possible with another Process@ session). So, in this mode, the “Start” and “Stop” acquisition buttons are deactivated. In this mode, it is no possible, too, to save experiments***.

- Once the experiment is loaded, click on “**Data / Variable**”, and select the variable that the user wants to check the data (as described in the previous section).

- Click on the right button of the mouse, select “**Copy all data to clipboard**”. Now these data can be pasted in any data sheet (Excel, Origin, Txt, etc). The format of the Excel columns must have the same format for each cell (number with/without decimals, date, time, etc.) as the data that have been imported.

- Once the data are recovered, exit this Process@ session. If the user wants to recover another old experiment, he has to open a new software session (Figure 5-4).

5.5.5 SAVING EXPERIMENT TEMPLATES

The templates generated for performing an experiment may be stored for subsequent recovery and use. Proceed as follows to store a template in the memory:

1. Stop acquisition
2. On the functions bar, select “**Experiment**”
3. Select the option “**Save Experiment as…**”.

A window will appear on the screen like the one shown in Figure 5-21, where the user may allocate a name to the template to be saved. Complete the saving process by clicking on “**Accept**”.

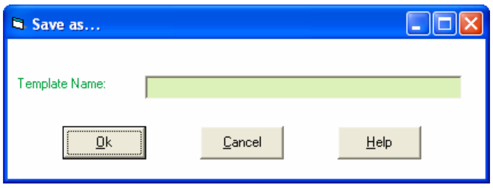


Figure 5-21

Saving an experiment template stores the following for subsequent use:

- The session configuration panel.
- The configuration of each one of the devices in the process control graphs.
- The acquisition time

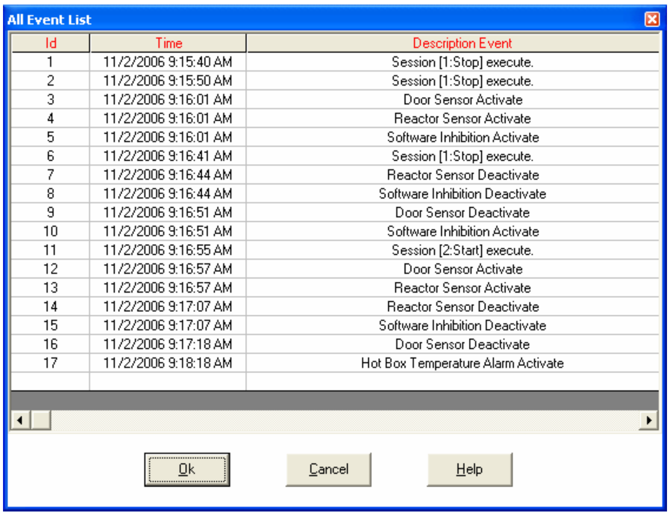
To recover a template stored in the memory, simply select the name of the required template when creating a new experiment (see section 5.4.1 in this manual).

5.5.6 CHECKING THE ALARMS

All events and alarms are recorded in a list that can be consulted by the user: select Data / Alatms, as is shown in Figure 5-22.

Figure 5-22

This list contains all the alarms and events (starting sessions, devices activation, etc) that have taken place during the acquisition time (Figure 5-23).



All Event List

Id	Time	Description Event
1	11/2/2006 9:15:40 AM	Session [1:Stop] execute.
2	11/2/2006 9:15:50 AM	Session [1:Stop] execute.
3	11/2/2006 9:16:01 AM	Door Sensor Activate
4	11/2/2006 9:16:01 AM	Reactor Sensor Activate
5	11/2/2006 9:16:01 AM	Software Inhibition Activate
6	11/2/2006 9:16:41 AM	Session [1:Stop] execute.
7	11/2/2006 9:16:44 AM	Reactor Sensor Deactivate
8	11/2/2006 9:16:44 AM	Software Inhibition Deactivate
9	11/2/2006 9:16:51 AM	Door Sensor Deactivate
10	11/2/2006 9:16:51 AM	Software Inhibition Activate
11	11/2/2006 9:16:55 AM	Session [2:Start] execute.
12	11/2/2006 9:16:57 AM	Door Sensor Activate
13	11/2/2006 9:16:57 AM	Reactor Sensor Activate
14	11/2/2006 9:17:07 AM	Reactor Sensor Deactivate
15	11/2/2006 9:17:07 AM	Software Inhibition Deactivate
16	11/2/2006 9:17:18 AM	Door Sensor Deactivate
17	11/2/2006 9:18:18 AM	Hot Box Temperature Alarm Activate

Ok Cancel Help

Figure 5-23

5.6 CONNECTION TO A GAS CHROMATOGRAPH

The Microactivity Reference unit is prepared for connecting to a gas chromatograph, actuating the sampling injection valve for starting the analysis.

Both devices (external alarm and chromatography) will be connected to the Microactivity-Reference using the wire “External Control”, provided by PID Eng & Tech with the unit:

- Connector "EXTERNAL CONTROL": BINDER 680 female 6 pin
  - PIN 1 : Chromatograph contact: BROWN
  - PIN 2 : Chromatograph contact: WHITE
  - PIN 3 : Emergency contact: YELLOW
  - PIN 4 : Emergency contact: GREEN
  - PIN 5 : Without cable
  - PIN 6 : Without cable

1. CONNECTION TO AN EXTERNAL ALARM

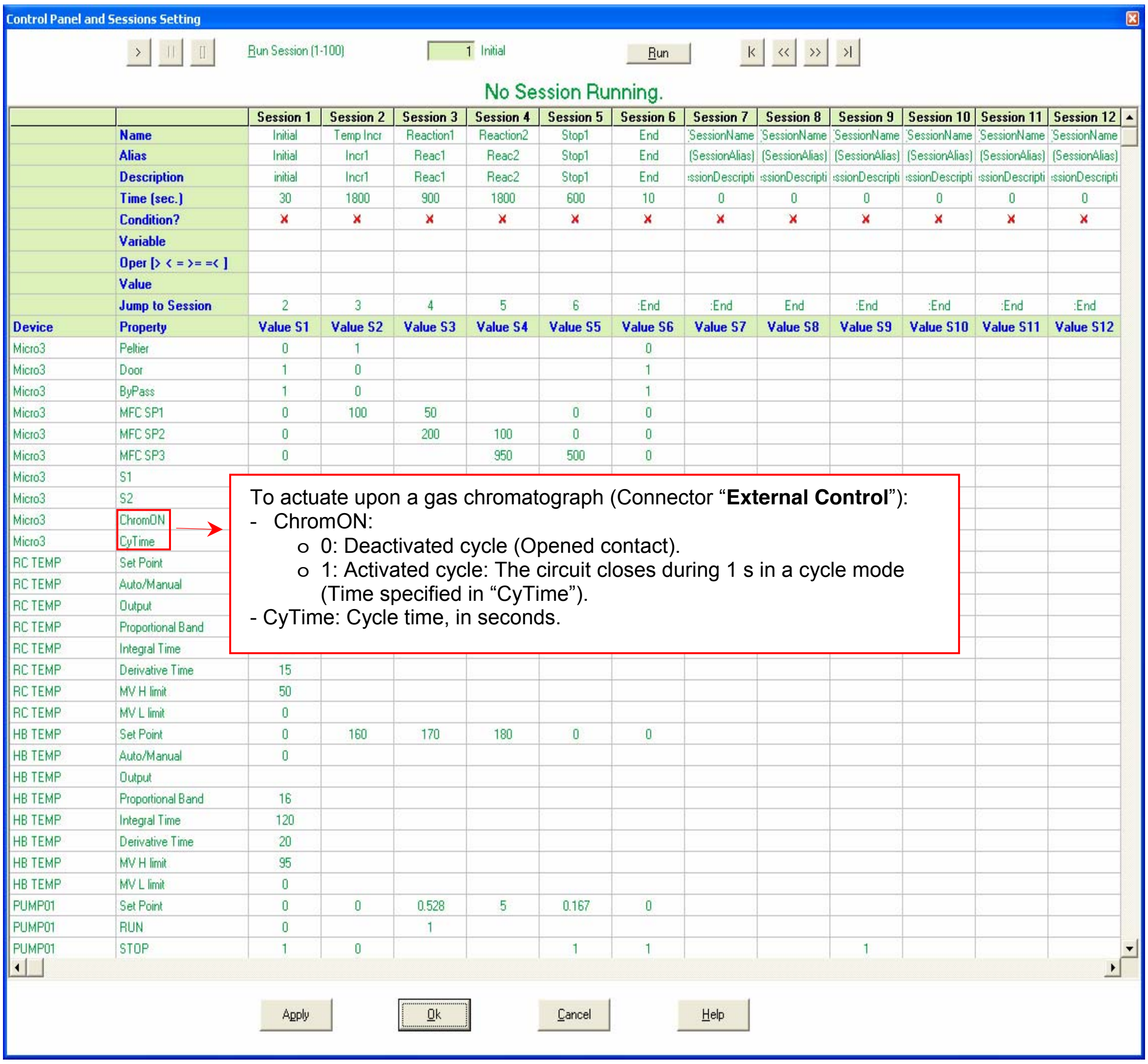
- The connection to an external alarm (gas detectors, etc.) has to be made with the wires yellow and green.
- The contact is normally opened: The two wires have to be connected to a relay that is opened normally and closes when the alarm activates.
- The actions that the unit makes when an external alarm appears are the following:

  - Reactor Furnace: OFF
  - Hot box Heaters: OFF
  - Inhibition Session: ON
  - Buzzer: ON

- The actions of the external alarm on the MFC and the pump of liquids are the same ones that the configured by the user in the touch screen for a pressure alarm
- When the external alarm disappears, the unit returns to the operation conditions.

2. CONNECTION TO A GAS CHROMATOGRAPH

- The connection to the chromatograph will be made with the cables brown and white.
- The MA-Ref unit has to be connected to the “Remote Control” connector of the GC.
- The contact is normally opened, closing itself during 1 second when a pulse is sent to the chromatograph.
- *Operation*: It is possible to activate a cycle to actuate upon a gas chromatograph in the Sessions Setting Panel of Process@ software. This cycle actuates closing the electrical circuit that connects with the GC during 1 second. The user can configure the cycle time (time between 2 closings) in the fill “CyTime” (Figure 5-24).
- Visualization: In the process screen is configured the device for checking the status of the chromatography cycle (Figure 5-25).



Control Panel and Sessions Setting

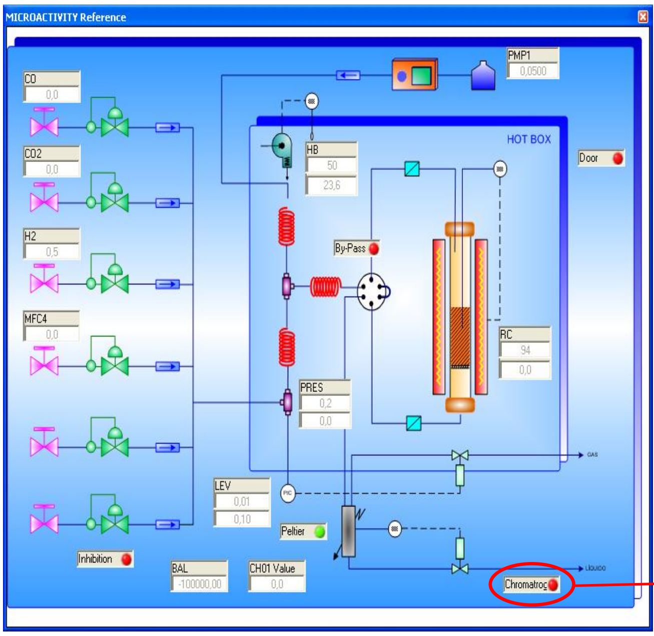
Run Session (1-100) 1 Initial Run

No Session Running.

		Session 1	Session 2	Session 3	Session 4	Session 5	Session 6	Session 7	Session 8	Session 9	Session 10	Session 11	Session 12
	Name	Initial	Temp Incr	Reaction1	Reaction2	Stop1	End	SessionName	SessionName	SessionName	SessionName	SessionName	SessionName
	Alias	Initial	Incr1	Reac1	Reac2	Stop1	End	(SessionAlias)	(SessionAlias)	(SessionAlias)	(SessionAlias)	(SessionAlias)	(SessionAlias)
	Description	initial	Incr1	Reac1	Reac2	Stop1	End	ssionDescripti	ssionDescripti	ssionDescripti	ssionDescripti	ssionDescripti	ssionDescripti
	Time (sec.)	30	1800	900	1800	600	10	0	0	0	0	0	0
	Condition?	×	×	×	×	×	×	×	×	×	×	×	×
	Variable												
	Oper [> < = >= =<]												
	Value												
	Jump to Session	2	3	4	5	6	:End	:End	End	:End	:End	:End	:End
Device	Property	Value S1	Value S2	Value S3	Value S4	Value S5	Value S6	Value S7	Value S8	Value S9	Value S10	Value S11	Value S12
Micro3	Peltier	0	1				0						
Micro3	Door	1	0				1						
Micro3	ByPass	1	0				1						
Micro3	MFC SP1	0	100	50		0	0						
Micro3	MFC SP2	0		200	100	0	0						
Micro3	MFC SP3	0			950	500	0						
Micro3	S1												
Micro3	S2												
Micro3	ChromON												
Micro3	CyTime												
RC TEMP	Set Point												
RC TEMP	Auto/Manual												
RC TEMP	Output												
RC TEMP	Proportional Band												
RC TEMP	Integral Time												
RC TEMP	Derivative Time	15											
RC TEMP	MV H limit	50											
RC TEMP	MV L limit	0											
HB TEMP	Set Point	0	160	170	180	0	0						
HB TEMP	Auto/Manual	0											
HB TEMP	Output												
HB TEMP	Proportional Band	16											
HB TEMP	Integral Time	120											
HB TEMP	Derivative Time	20											
HB TEMP	MV H limit	95											
HB TEMP	MV L limit	0											
PUMP01	Set Point	0	0	0.528	5	0.167	0						
PUMP01	RUN	0		1									
PUMP01	STOP	1	0			1	1			1			

Apply Ok Cancel Help

Figure 5-24



STATUS	GC Cycle
	Activated
	Deactivated

Figure 5-25

6. PERFORMING CATALYTIC TESTS

6.1. LOADING THE CATALYTIC BED IN THE REACTOR

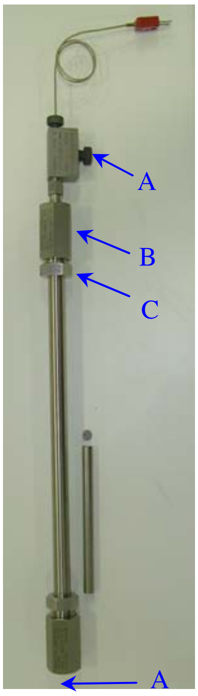


Figure 6-1

Insert the catalytic bed inside the reactor by proceeding as follows:

1. Open the reactor hot box by pressing the "**Door**" key on the touch screen.
2. Disconnect the reactor's thermocouple (red pin). This will trigger the system's temperature alarm, which can be deactivated on the touch screen's alarm panel (see section 4.5.1 in this manual).
3. Using a spanner, release the reactor at connections "A" (see Figure 6-1).
4. Hold the reactor with a clamp on the upper part "B" and loosen that connection by exerting pressure on "C".
5. Place the reactor in a vertical position, unscrew "B" and remove the thermocouple from the reactor.
6. Empty the reactor and flush with compressed air through the lower end, in the opposite direction to the gas flow inside.
7. Insert the catalyst through the upper end of the reactor, with a particle size greater than 10 μm (a small quantity of quartz wool may be inserted beforehand, to avoid the passage of fine particles through the porous plate) and, if considered necessary, pack the reactor with carborundum up to 2-3 cm below the upper end in order to avoid the dead volume.
8. Clean threads "B" and "C", insert the thermocouple inside the bed, close the reactor and reconnect the gas inlet and outlet lines by means of connections "A".
9. To avoid a heating peak in the reactor oven, switch off the Microactivity-Reference before plugging the thermocouple into the hot box. Then switch on the equipment again.
10. Perform a *Leaks test*: See section 7.1.1 of this manual.

6.2. PROGRAMMING A SEQUENCE OF EXPERIMENTS

For programming a sequence of experiments, proceed as outlined in section 5.4 of this manual: "Performing an experiment".

In general terms, the sessions that constitute the experiment should be configured as follows:

- An initial session corresponds to the secure shutdown of the system.
- The second session takes the system to the operating conditions under which the experiment is to be performed (e.g.: heat the reactor up to operating temperature). The duration of this session is to be sufficient to allow the system to attain stable operating conditions, which may be carried out by setting long session times or else by using the condition evaluation function.
- Subsequent sessions correspond to the operating conditions in which catalytic measurements are to be taken.
- The final two sessions correspond to sessions involving the secure shutdown of the system, with the last one being the same as the session programmed as no. 1

7. MAINTENANCE OF THE EQUIPMENT

7.1. WEEKLY MAINTENANCE

7.1.1 LEAKS DETECTION

It is necessary to perform a leaks test when:

- The user opens the reactor for replacing the catalyst bed
- The user detects a bad control in pressure control

For checking leaks in the unit, proceed as follows:

- Put the pressure controller in manual mode, totally opened (100%).
- Put the level controller in manual mode, totally closed (0%). Once the system is at working pressure, it would be necessary to check if this valve is totally closed or if it is necessary to recalibrate the zero point.
- Put a tap in the gas outlet on the hot box and introduce an inert gas flow in the system (He, N_2, etc.). Put the unit at habitual working pressure.
- Close the gas inlet, as well as the system on/off stopcock for gas and verify that the pressure in the system remains constant over a period of time. If this is not the case, use a soapy solution to locate possible leaks (subsequently dry the entire system).
- When the leaks has been detected and eliminated, clean the equipment, removing the soapy solution.

7.1.2 CLEANING THE UNIT

For clearing the external of the unit, use a wet-cloth with water for preventing dust accumulation.

For internal cleaning, it can be used water or any solvent (alcohol, acetona, etc.) for eliminate soapy solution rest.

In case of cleaning with any solvent, the user has to manage properly the generated residues, attending to its nature.

7.2. MONTHLY – QUARTERLY MAINTENANCE

Depending on the use of the reactor and the reaction products the user should make a monthly or quarterly maintenance, consisting on:

7.2.1 REPLACING THE FILTERS POROUS PLATES

The reactor is provided with two 10 microns porous plates that could get blocked as a consequence of the continuously use. For replacing them, take out the filters from the unit, open the filters body and remove the 10 microns porous plate. Replace it for a new one, supplied as an equipment spare part (Ref. Vici Valco – 10FR4)

If, as a consequence of this maintenance, the filter body has leaks, it is recommended to replace it by another one: Vici Valco, VA_ZBUFR2F10.

Figure 7-1

The replaced porous plate could be contaminated with hydrocarbons or other kind of residues. The user of the unit must manage these residues, asking to an authorized manager and attending to the environmental policy of the laboratory where it is being used.

7.3. ANNUAL OR LATER MAINTENANCE

7.3.1 REPLACING THE FUSE

The Microactivity-Reference has incorporated a 3 A fuse at the rear of the reactor for protecting the power sockets. For replace it (Figure 7-2), turn the equipment off (put the circuit breaker in the "OFF" position) and replace it by another one with the same characteristics: 3 A – 250 V.

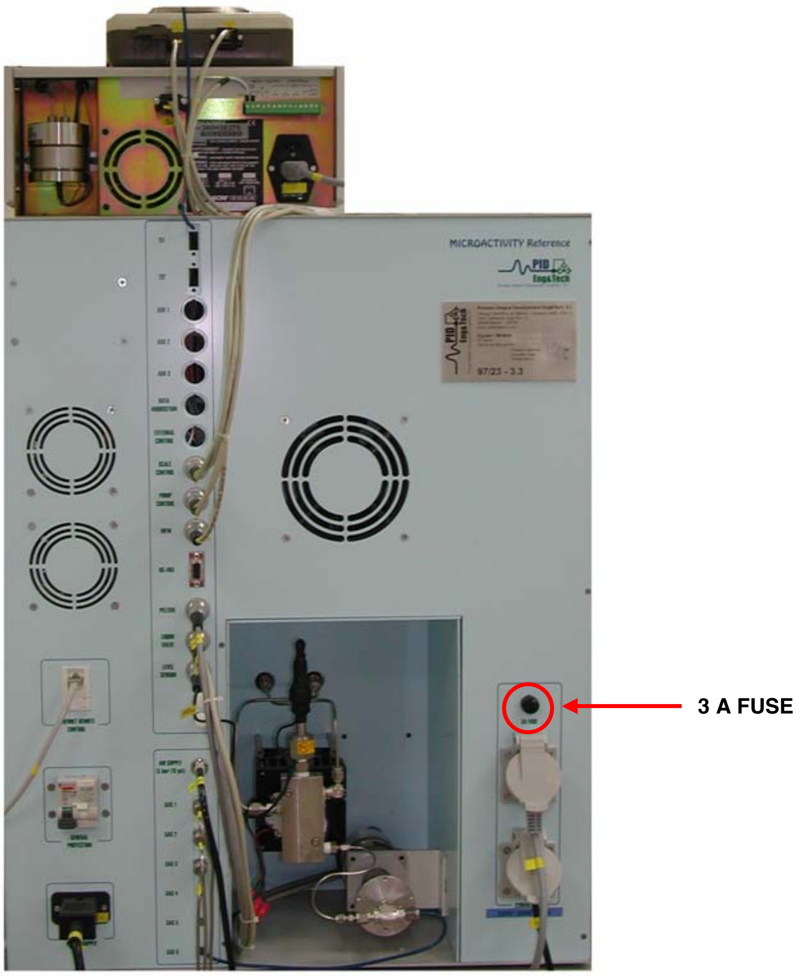


Figure 7-2

7.3.2 REPLACING THE KALRETZ SEALS IN THE GAS MIXER

If the unit woks with high-corrosive gases, it should be convenient to replace annually or later the kalretz seals of the check valves, before the gas mixer: Open the valves body and replace the elastomeric seal by another one (same model and material): contact with Process Integral Development Eng & Tech Technical Service.

The replaced o-ring could be contaminated by any corrosive gas. The user of the unit must manage these residues, asking to an authorized manager and attending to the environmental policy of the laboratory where it is being used.

7.3.3 REPLACING THE REACTOR POROUS PLATE

Due to a continued use of the unit with high viscosity liquids or substances that generate solid deposits, the porous plate of the reactor could get blocked, identifying this fact by an increase of the pressure of the system. In this case, the user must contact with the Process Integral

Development Eng & Tech Technical Service to substitute it by another plate of the same characteristics, or of higher porosity.

The replaced porous plate could be contaminated with hydrocarbons. The user of the unit must manage these residues, asking to an authorized manager and attending to the environmental policy of the laboratory where it is being used.

7.3.4 REPLACING A MASS FLOW CONTROLLER

The replacement of one or more MFC may be caused by:

- Their faulty operation: in this case, the user is to verify that this performance is not due to an unsuitable gas inlet pressure (consult the specifications of the MFC that are included in the equipment's documentation) or to an incorrect configuration of the MFC on the touch screen (see section 4.5.2 in this manual).
- Changes in the user's requirements, insofar as the type of inlet gas or the flow supplied is concerned.

The steps to be followed for replacing a MFC are as follows:

1. Switch off the Microactivity-Reference unit and switch the main circuit breaker to OFF.
2. Unscrew and remove the right-hand side panel on the Microactivity-Reference
3. Locate the MFC that is to be replaced (see Figure 7-3), disconnect the control cable, unscrewing the unit's lower panel and releasing the end connections.

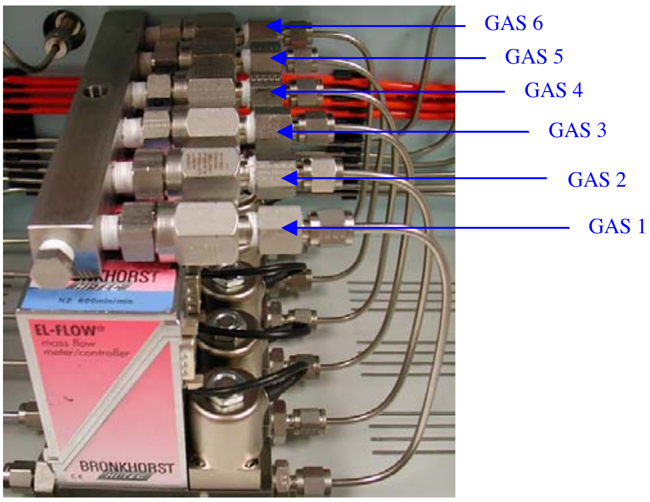


Figure 7-3

4. The MFC that are installed in the equipment are to have the same characteristics as the one being replaced (check the specifications of the MFC that are enclosed in the documentation):

   - Mass Flow Controllers, HI-TEC by BRONKHORST, model EL-FLOW.

   - IDENTIFICATION No.**: F_211C_FAC_11V** (Figure 7-4):

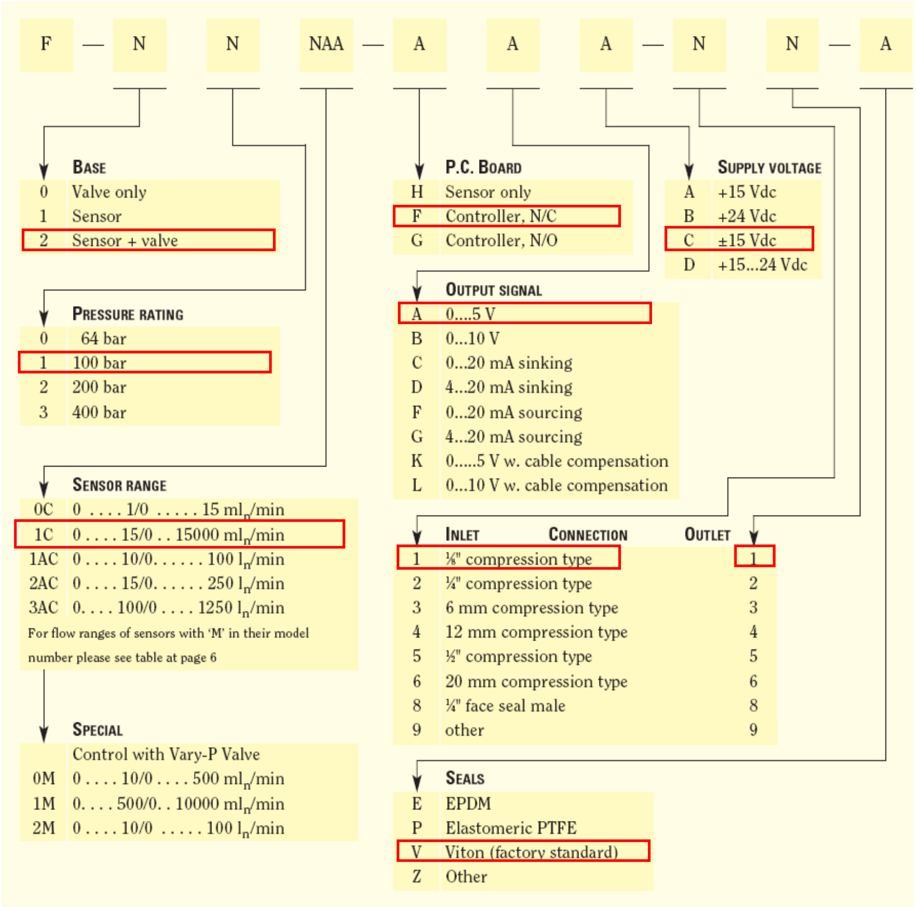


Figure 7-4

- Gas: Determined by the user
- Maximum flow: Determined by the user
- Inlet pressure: Determined by the user

5. Place the new MFC in the distributor, screwing it in from the lower panel and attaching the end connectors (it is advisable to attach a label indicating the gas it contains and its operating flow). Place it in such a way that the arrow is pointing to the right (indicating the direction of gas flow).
6. Connect the control cable to the MFC.
7. Screw the right-hand side panel back onto the Microactivity-Reference unit.
8. Switch on the unit.
9. Configure the MFC installed on the touch screen of the Microactivity-Reference unit:
   - Pressing "**F1**" on the touch screen grants access to the main menu (CONFIG SETUP).

Figure 7-5

- Press on the field "*MASS FLOW SETUP*": The configuration menu for the mass flow controllers will be displayed:

Figure 7-6

- Configure the fields of the new MFC:

- Maximum flow.
- Units in which the gas flow is expressed.
- Name of the gas.
- % of alarm: Deviation alarm. This alarm is inhibited during the time specified in the *Delay Time Alarm* (in sec.), operating if during this time the specified deviation is maintained with respect to the set value.

These parameters are modified by pressing on their corresponding yellow boxes and entering the new values by means of the keys that are displayed on screen. Before beginning to work with the equipment, it is important to ensure that each reactant gas MFC installed has been properly set up, and that all the other MFC's not installed have their fields set to zero.

Press the "**Exit**" key to return to the main menu.

8. EUROPEAN DIRECTIVES

1. Directive 97/23/EC: *Pressure Equipment*

The plant complies with European Directive 97/23/EC and Spain's Royal Decree 769/1999 that lays down the provisions for the application, in Spain, of said directive, regarding the design, manufacture and evaluation of compliance of pressure equipment and equipment subject to a maximum allowable pressure PS exceeding 0.5 bar.

The plant is supplied with Markings and Statement of Compliance as per article 3, section 3 of European Directive 97/23/EC and Spain's RD 769/1999.

2. Directive 94/9/EC: *Equipment and protective systems intended for use in potentially explosive atmospheres*

The plant is not to be used in potentially explosive atmospheres.

Directive 94/9/EC (on the approximation of the laws of the member states concerning equipment and protective systems intended for use in potentially explosive atmospheres) in its chapter I, Article 1, section 4, lays down that:

"The following are excluded from the scope of this Directive: (…) Equipment intended for use in domestic and non-commercial environments where potentially explosive atmospheres may rarely be created, solely as a result of the accidental leakage of fuel gas…"

The guidelines on the application of Directive 94/9/EC, of May 2000, state in their section 4.1.2. a) that:

"Equipment is only considered to be within the scope of the directive it if is intended (either in whole or in part) to be used in a potentially explosive atmosphere; the fact that an intended potentially explosive atmosphere might be present inside the equipment is not relevant..."

Furthermore indicating:

"Products that are not designed for use under atmospheric conditions (1) do not fall within the sphere of application of Directive 94/9/EC, even when an explosive atmosphere may form under atmospheric conditions during start-up, disconnection or maintenance. This would form part of risk assessment on the part of the user and could lead to the specification of ATEX apparatuses for installation of a near-by container".

(1) Directive 94/9/EC does not define atmospheric conditions. The relevant standards indicate a temperature range of –20 °C to 60 °C and a range of pressure between 0.8 bar and 1.1 bar as a basis for design and intended use of products.

Consequently, the Microactivity-Reference unit is not designed for operating under potentially explosive atmospheres, but as a result of improper use of the unit or a lack of maintenance of the same, the unit could generate a potentially explosive atmosphere.

It is the responsibility of the end user to assess the risks, implement suitable safety and protective measures, as well as locate the equipment in special laboratories with inflammable gas

detectors in order to reduce to a minimum the risks stemming from operation of the equipment. The Microactivity-Reference unit caters for connection to an external alarm that would trigger the unit's secure shutdown.

3. **Directive 98/336/EEC: Electromagnetic compatibility**

The Microactivity-Reference unit, as per Directive 89/336/EEC of 3 May 1989, is considered to be equipment that may cause electromagnetic disturbances or whose operation may be affected by said disturbances, given that it is a piece of equipment or installation that contains electrical and/or electronic components. It is therefore to be constructed in such a manner that:

The electromagnetic disturbances generated are limited to a level that enables the apparatus to operate in accordance with the purpose for which it was designed.

The apparatus has a suitable level of intrinsic immunity (ability to operate without detriment to quality in the presence of a magnetic disturbance) that enables it to operate in accordance with the purpose for which it was designed.

Figure 8-1

The Microactivity-Reference unit complies with Directive 98/336/EEC, having passed all electromagnetic compatibility tests required by the same (Figure 8-1 shows pictures of the Microactivity-Reference unit in the anechoic chamber, where part of said tests were carried out).

9. ENVIRONMENTAL POLICY

Process Integral Development Eng & tech bakcs the environment, focusing its activities towards the minimization of the impacts to surroundings, with the commitment from management to follow the principles included inside the policy.

The different devices and operations with relevant environmental injuries have been described in this manual (porous plates, filters, o-rings, solvents and main board battery). PID Eng & tech ask the final user to be responsible with the environment, following the actions specified in the chapter 8 “Maintenance of the equipment” for the disposal of toxic or dangerous wastes, and attending to the environmental policy in force in the laboratory or company where the equipment is working.

If the user wishes to get rid off of the unit, he must hand it in to an authorized manager or ship it to Process Integral Development Eng & tech, where it will receive the proper treatment.

10. REACTOR TROUBLESHOOTING

SYMPTOM	POSSIBLE CAUSES	SOLUTION
The equipment does not switch on	The switch on the front is in the off position.	Press the switch on the front.
	The main circuit breaker on the rear is off.	Switch the main circuit breaker to "ON".
	The power supply to the equipment has not been properly connected.	Verify the correct connection of the power supply that is on the rear of the equipment.
	The system's fuse has blown.	Open the right-hand side of the equipment and replace the fuse on the board (contact the distributor).
No communication is established between the reactor and the control PC	The Ethernet connection has not been properly installed (wrong network cable, incorrect operation of the switch, wrong IP address).	Check that the Ethernet connection is working properly and verify the system's IP address ("MISC" Menu on the touch screen). Verify the configuration of the PC's local area network. Reboot both systems.
There is no communication between the reactor and the touch screen	Blocking of the microprocessor.	Reboot the equipment. If the problem persists, please contact the distributor.
The door on the reactor does not open and/on the by-pass valve does not operate	There is no air pressure in the system.	Check the gas installation and make sure the system's air inlet pressure is 5 bar.
	Leaks in the compressed air lines or poor coupling of the pneumatic connections.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the right and left-hand side panels on the equipment and check the orange polyurethane pipes.
No gases are entering the equipment	No gas pressure reaches the equipment.	Check the gas installation and make sure the gas cylinders are open.
	The on/off stopcocks on the front panel are off.	Open the on/off stopcocks.
	A system alarm has been triggered that has shutdown the gas inlet.	Check the alarm and reset it (providing the situation that triggered it has been resolved).
	The pressure in the system is close to the pressure in the gas cylinders.	Increase the pressure on the gas inlet (see the specifications of the MFC) or lower the operating pressure.
The flow of one of the gases varies without keeping to the set-point.	The cable that connects the MFC to the board has been badly connected (loose).	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the right-hand side panel on the equipment and check the connection of the MFC cables.
A reading is recorded on one of the MFC that has not been installed (noise).	A maximum flow has been set on the MFC configuration panel on the touch screen for one of the gases that has not been installed.	Zero set all the fields for those MFC that have not been installed on the equipment.
The gas flow does not reach the set–point established, remaining stable at a lower value.	There is no communication between the touch screen, the control PC and the equipment hardware.	Reboot the equipment.
	Insufficient gas pressure is reaching the equipment.	Verify that the gas inlet pressure on the equipment exceeds the operating pressure
	The set-point established is below 10% of the maximum flow of the mass flow controller.	It is not advisable to operate below 10% of the maximum flow of the MFC: Replace the controller with another that is suited to the process requirements.
	Modification has been made of the maximum gas flow established on the touch screen for the configuration of the mass flow controllers.	Reset the maximum flow value for each one of the controllers (see the specific documentation for each MFC).

The Gilson liquid pump does not switch on, with the Microactivity switched off.	The pump is connected to the 220V sockets on the rear of the equipment.	These sockets are not energised when the equipment is switched off: plug the pump into another socket (separate from the unit) or switch on the equipment.
The Gilson liquid pump does not switch on, with the Microactivity switched on.	The 3A-250V fuse in the fuse-box on the rear panel has blown (or is missing).	Change the fuse.
	The pump's electrical installation has not been performed correctly.	See the section "Electrical installation" in this manual.
	The on/off switch is in the (0) position.	Turn the switch on the rear of the pump to the (I) position.
The Gilson pump does not register the information or the set-points relayed to it from the Process@ software.	If a prior manual control has been made on the pump's display, it is not possible to establish digital communication with the PC, and vice-versa.	Reboot the pump. If this is not possible, control of the same is to be maintained in manual mode.
The Gilson pump does not respond to the parameters that are manually entered into the display.	If a prior digital communications control has been made between the PC and the equipment, it is not possible to perform manual control on the display, and vice-versa.	Reboot the pump. If this is not possible, control of the same is to be maintained by digital communications, through the Process@ software.
No liquid is entering the reactor or the flow is unstable (Pressure of the head below operating pressure)	No backpressure has been installed and operation is at atmospheric pressure.	There needs to be a pressure of 10-15 bar in the pump head: - Install a backpressure prior to the inlet on the liquid non-return valve. - Only feed liquids when the operating pressure is above 10-15 bar.
	The pump has not been properly vented.	Vent the pump, releasing the liquid inlet line on the reactor.
	There is a leak in the system's liquid inlet.	Verify all the liquid inlet lines on the system.
	An alarm has been triggered in the system that has shutdown the system's liquid inlet.	Check the alarm and reset it (providing the situation that triggered it has been resolved).
No liquid is entering the reactor (Pressure of the head close to operating pressure)	No backpressure has been installed and operation is at atmospheric pressure.	There needs to be a pressure of 10-15 bar in the pump head: - Install a backpressure prior to the inlet on the liquid non-return valve. - Only feed liquids when the operating pressure is above 10-15 bar.
	There is no liquid in the pump tank.	Fill the tank with the reactant liquid.
	The 3-port valve on the pump is not in the normal operating position (injection from the tank).	Set the valve to the suitable position (see the section "Venting the pump" in this manual or in the pump manual).
	An alarm has been triggered in the system that has shutdown the system's liquid inlet.	Check the alarm and reset it (providing the situation that triggered it has been resolved).
No liquid is entering the reactor (Pressure of the head significantly above operating pressure)	A blockage of solid deposits has formed in the evaporator.	Replace the evaporator.
	Faulty operation of the liquid non-return valve.	Verify the status of the non-return valves and, if necessary, replace.
	An alarm has been triggered in the system that has shutdown the system's liquid inlet.	Check the alarm and reset it (providing the situation that triggered it has been resolved).

There is no temperature reading in the reactor bed.	The reactor's thermocouple has not been connected properly.	Verify the thermocouple's connector inside the hot box.
	The reactor's thermocouple is not working properly.	Replace the reactors' thermocouple with another of identical characteristics.
	Poor connection of the thermocouple's damping cable on the 18-pole wire housing.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the left-hand side panel on the equipment and check the connections to the 18-pole wire housing.
The reactor's temperature reading is incorrect	The TOHO reaction temperature controller has been configured incorrectly.	Introduce the factory-set default parameters in the controller. If these are not known, please contact the distributor.
The reactor's temperature controller is not working properly.	The controller's PID parameters have been modified.	Restore the optimum control parameters (see the section "The controllers" in this manual).
	Control mode has been set to "manual" or "Rdy", instead of to "automatic".	Set to automatic (Run) in the controller's _nd parameter.
	An alarm has been triggered in the system that has shutdown the reactor oven (oven not fully closed, door open on hot box).	Check the alarm and reset it (providing the situation that triggered it has been resolved).
The electric heater on the hot box does not operate.	The parameter _EH1 on its controller has been modified. (Temperature above which it starts operating).	Re-set the value of parameter _EH1 to 40 ºC on the hot box's temperature controller (SET 2 menu on the controller).
	The hot box's temperature controller has been set to "manual", at 0%.	Set the control mode to automatic (Run) on the controller's _nd parameter.
	An alarm has been triggered in the system that has shutdown the heating on the hot box	Check the alarm and reset it (providing the situation that triggered it has been resolved).
	The heater is not working properly	Replace the heater (contact the supplier).
The turbine does not operate correctly (it does not turn properly or it makes an odd noise)	The forward blades on the turbine are rubbing against the ceramic cable housing or the heating cartridges.	With the equipment disconnected, remove the front cover on the turbine inside the hot box (it clips off) and remove the cabling.
	The rear blades on the turbine for cooling the motor are rubbing against the turbine mount.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the left-hand side panel (or the upper one, if necessary) and adjust the motor's cooling blades.
The hot box's temperature controller is not working properly.	The controller's PID parameters have been modified.	Restore the optimum control parameters for this controller (see the section "The controllers" in this manual).
	An alarm has been triggered in the system that has impeded the heating of the hot box (door open on the hot box)	Check the alarm and reset it (providing the situation that triggered it has been resolved).
	Control mode has been set to "manual" or "Rdy", instead of to "automatic".	Set the controller's _nd parameter to automatic (Run).
There is no reading of the system's pressure in the controller.	The pressure sensor is not connected to the board.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the right-hand side panel and verify the sensor's connection.
	The TOHO pressure controller is not connected to the board.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the right-hand side panel and verify the TOHO pressure connection.
	The power source +15/-15/+5 is short-circuiting (power LED blinking) or is faulted (LED off). If this occurs, there will be no level reading, if the equipment is fitted with this option.	Verify the status of the source by unscrewing the right-hand side (equipment disconnected and without power supply) and observe the LED. If it is blinking, disconnect the controllers on the rear panel one by one, until the one causing the short-circuit is located (the LED will stop blinking).
The pressure in the system rises	The pressure controller is set to "manual", fully closed (0%).	Set to automatic (Run) in the controller's _nd parameter.

uncontrollably without responding to the set-point, with the 3-port valve in the "by-pass" position.	A blockage has formed in the gas outlet line, outside the reactor box (on the way to the analysis system).	Release the joint between the system's gas outlet line and the reactor box (external left-hand side of the reactor) and see whether the pressure decreases. If it does, clean or replace the blocked section. If it does not, continue by verifying the next cause.
	The needle on the micrometric pressure regulation valve has become stuck at zero.	Release the joint between the gas line and the regulating valve and see whether the pressure decreases. If it does, replace the regulating valve (notify the distributor).
	The valve's zero-setting has been modified	Check and adjust the zero-setting on the pressure control valve (see the section "Configuration of the V4.0 Servo Digital Unit" in the manual).
	A blockage has formed on the liquid – gas separator inlet and/or outlet lines.	Release the inlet line to the separator and see whether the pressure decreases. Clean the inlet and outlet lines and the condenser tank with ethanol + compressed air. Otherwise, continue by assessing the next possible cause.
	Blockage in the by-pass valve or in the gas preheating and/or mixer lines	Release each one of the sections of pipe and see whether the pressure falls. Clean and/or replace the blocked section.
No rise in pressure in the system	No gases are entering the system	Check the symptom "No gases are entering the equipment".
	Leak in the reaction system	Perform a leak test on the equipment (See the section "Performing catalytic tests" in this manual).
The pressure in the system rises uncontrollably without responding to the set-point, with the 3-port valve in the reaction position.	Set the 3-port valve to the by-pass position. If the pressure in the system does not decrease, check all possible causes of the prior symptom: The pressure in the system rises uncontrollably without responding to the set-point, with the 3-port valve in the "by-pass" position."	
	The filter on the reactor outlet is blocked.	Release the gas outlet line where it joins the filter and see whether the pressure decreases. If it does, replace the filter. If it does not, continue by verifying the next cause.
	The reactor's porous plate is blocked.	Release the connecting joint between the gas inlet line and the reactor and see whether the pressure decreases. If it does, empty the reactor and clean the plate by flushing it with compressed air counter-current to the gas flow. If this is not the solution, replace the porous plate (inform the distributor).
	The filter on the reactor gas inlet is blocked.	Release the gas outlet line before it passes through the filter and see whether the pressure decreases. If it does, replace the filter. If it does not, continue by verifying the next cause.
The pressure in the system varies, without stabilising at the set-point.	The controller's PID parameters have been modified.	Restore the optimum control parameters for this controller (see the section "The controllers" in this manual).
	Incorrect operation of the level control system in the separator (if operating with this option).	Stop feeding liquids into the system and close the level control valve. See whether the pressure stabilises.
	Leak in the reaction system	Perform a leak test on the equipment (See the section "Performing catalytic tests" in this manual).
	The valve's zero-setting has been modified	Check and adjust the zero-setting on the pressure control valve (see the section "Configuration of the Servo Digital V4.0 unit" in the manual).
The pressure in the system does not reach the set-point, stabilising at a lower value.	Insufficient gas pressure is reaching the equipment.	Make sure the gas inlet pressure on the equipment exceeds the operating pressure.
	Leak in the reaction system	Perform a leak test on the equipment (See the section "Performing catalytic tests" in this manual).

The sensor's reading exceeds 2 ml or is above zero with the tank empty.	The maximum and minimum levels of oscillation set in the calibration of the sensor have been modified.	On the level sensor's touch screen, restore the values obtained in calibration. If these are not available, re-calibrate the sensor (see the section "Calibrating the level sensor" in this manual).
	The sensor is dirty.	Dismantle the condenser and the sensor and clean them with ethanol + compressed air.
There is no reading from the level sensor in the separator, with liquid in the condenser.	The sensor has not been properly connected.	Check and properly adjust the sensor connection on the rear of the equipment.
	Incorrect controller configuration	Restore the controller to its original parameters.
	The sensor is not working properly	Replace the level sensor (contact the distributor).
	The level sensor is not connected to the board.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the right-hand side panel and verify the sensor's connection.
	The TOHO level controller is not connected to the board.	Disconnect the equipment (switch the circuit breaker to OFF), unscrew the right-hand side panel and verify the TOHO level connection.
	The power source +15/-15/+5 is short-circuiting (power LED blinking) or is faulted (LED off). If this occurs, there will be no level reading, if the equipment is fitted with this option.	Verify the status of the source by unscrewing the right-hand side (equipment disconnected and without power supply) and observe the LED. If it is blinking, disconnect the controllers on the rear panel one by one, until the one causing the short-circuit is located (the LED will stop blinking).
The sensor's reading exceeds 2 ml or is above zero with the tank empty.	The maximum and minimum levels of oscillation set in the calibration of the sensor have been modified.	On the level sensor's touch screen, restore the values obtained in calibration. If these are not available, re-calibrate the sensor (see the section "Calibrating the level sensor" in this manual).
	The sensor is dirty.	Dismantle the condenser and the sensor and clean them with ethanol + compressed air.
The level reading in the sensor does not remain stable at the set-point.	The controller's PID parameters have been modified.	Restore the optimum control parameters for this controller (see the section "The controllers" in this manual).
	Control mode has been set to "manual" or "Rdy", instead of to "automatic".	Set the controller's _nd parameter to automatic (Run).
	The sensor is dirty.	Dismantle the condenser and the sensor and clean them with ethanol + compressed air.
	The valve's zero-setting has been modified	Check and adjust the zero-setting on the level control valve (see the section "Configuration of the Servo Digital V4.0 unit" in the manual).
	The liquids pump is not working properly.	Check that the pump is working properly, providing the system with a constant stream of liquid.
No liquids are evacuated from the system (pressurised system in which the liquid level rises continuously without responding to its set-point)	The level controller is set to "manual", fully closed (0%).	Set the controller's _nd parameter to automatic (Run).
	A blockage has formed in the liquid outlet line, prior to its collection in the balance.	Release the liquid outlet line just after it passes through the micrometric valve and see whether any liquid is coming out of the system. If it is, replace the liquid outlet line.
	The needle on the micrometric level regulation valve has become stuck at zero.	Release the liquid outlet line on the separator and see whether liquid is coming out of the tank. If it is, replace the regulating valve (inform the distributor).
	The valve's zero-setting has been modified	Check and adjust the zero-setting on the level control valve (see the section "Configuration of the Servo Digital V4.0 unit" in the manual).

The change in programmed session does not occur.	The inhibition function has been triggered, as a safety measure in response to a system alarm.	This function is automatically shutdown when the situation triggering the alarm has been resolved, except in the case of pressure alarms. In this case, check the alarm and reset it (if the situation that triggered it has been resolved).
When saving a Process@ template ("Save session as …") no display is made of the screen for allocating the name to the template.	The program is still acquiring data from the equipment ("RUN" status).	In order to save a template, the system cannot be acquiring data from the unit: press the "STOP key".